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RAY T. BAYLESS, Editor

Vol. XXV

December, 1937

No. 4

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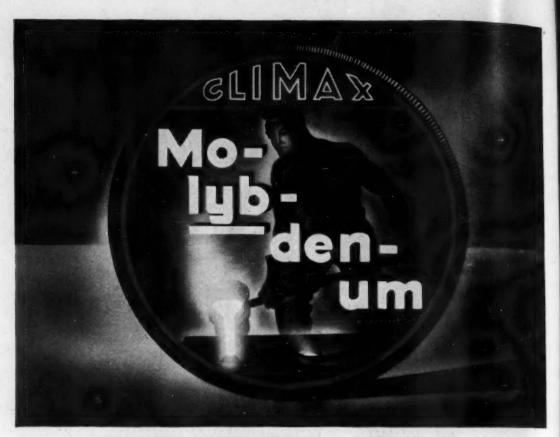
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PRACTICAL DATA

Years of shop and laboratory experience with alloy irons and steels have provided Climax with a great accumulation of valuable data. Parts of these data which have been published are doubtless familiar to members of the A. S. M. Much which remains in unpublished form constitutes a valuable source of information for any one interested in the development of Molybdenum steels and irons or in their selections for particular applications. These data and the services of our laboratory are at the service of those with specific ferrous problems. Climax Molybdenum Company, 500 Fifth Ave., New York City.

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STRUCTURAL AND HARDENING CHARACTERISTICS OF SOME IRON-COBALT-TUNGSTEN ALLOYS

By Wesley P. Sykes

Abstract

In the binary iron-tungsten alloys the iron-rich alpha solid solution exists in the body-centered cubic form at all temperatures below the freezing point. By substituting cobalt for part of the iron the alpha solid solution is replaced by the face-centered cubic gamma solid solution existing above about 950 degrees Cent. (1740 degrees Fahr.). This solid solution is subject to grain refinement during rapid cooling from above 950 degrees Cent. (1740 degrees Fahr.) in much the same fashion as is austenite in steel. Due to the relatively low diffusion rate of tungsten, however, no precipitation of the tungsten-rich phase (corresponding to cementite in steel) occurs until the quenched alloy is reheated to at least 300 degrees Cent. (570 degrees Fahr.). By reheating or aging at a temperature of 600 degrees Cent. (1110 degrees Fahr.), the hardness is increased from C38 to C65 within 15 minutes and reaches C69 in about one hour.

If the gamma solid solution is made to decompose isothermally at temperatures below 925 degrees Cent. (1700 degrees Fahr.), the resulting structures resemble very closely those observed in steel after similar treatment although the temperature ranges for the ternary alloy are some 300 degrees Cent. (570 degrees Fahr.) above those producing the corresponding structures in plain carbon steels. In the case of the 30 per cent cobalt, 20 per cent tungsten alloy, the structures resulting from isothermal decomposition of gamma at temperatures above 600 degrees Cent. (1110 degrees Fahr.) are of the lamellar or pearlitic type. At temperatures below 500 degrees Cent. (930 degrees Fahr.) the acicular type of structure results, while at about 550 degrees Cent. the decomposition does not begin within a period of 100 hours.

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Small amounts of additional alloying elements such as nickel, chromium, or vanadium retard the decomposition of the gamma solid solution so that in some instances it is almost completely preserved at room temperatures.

This is the Twelfth Edward DeMille Campbell Memorial Lecture, presented by Wesley P. Sykes, metallurgist, Cleveland Wire Works, General Electric Co., Cleveland. The lecture was presented at the Nineteenth Annual Convention of the Society, held in Atlantic City, October 18 to 22, 1937.

N this occasion, dedicated to the memory of Edward De Mille Campbell and to his achievements, it seems fitting to discuss a subject intimately allied with one of Professor Campbell's most active interests.

After the manner of the true scientist, Professor Campbell sought the explanation for the hardening of steel in the fundamentals of physical chemistry. Quite recently one of these basic principles has been properly assigned a leading role in the drama of modern metallurgy. I refer here to "the rate of reaction" which, expressed in one or another form, has been so brilliantly applied by Davenport and Bain to explain the hardening of steel.

Among the publications of Professor Campbell appears the title "Notes as to Rates of Reaction in Certain Steels at 930 Degrees Cent" (1). A brief quotation from this paper will suggest the basic nature of his investigations and his methods of analysis.

"The rate at which reactions take place in recently hardened steel at room temperature and at 100 degrees Cent. composed the subject matter of an article presented to the Iron and Steel Institute in 1918. The results obtained at that time show very strikingly the great influence of even a comparatively slight increase in temperature on the rate of reaction, since at 100 degrees Cent. (212 degrees Fahr.) there was a greater change in specific resistance in twelve hours than there was in two years at room temperatures." And again from the same paper—"In complex steels containing two or more elements other than carbon, such as chromium, tungsten or molybdenum, changes in (chemical) constitution due to differences in rate of cooling do not manifest themselves as simply as they do in the case of plain carbon steels."

As the subject for today's discussion, we have selected "The Structural and Hardening Characteristics of Some Iron-Cobalt-Tungsten Alloys." In these alloys the progress of reactions may be followed by observing the accompanying changes in microstructure, hardness and volume. While in these materials the carbon content is below 0.01 per cent (unless otherwise stated), in structure and in hardening behavior they resemble the carbon steels and especially the alloy steels.

Rogers and Seljesater (2) in 1932 first reported the remarkable changes in the hardening behavior of iron-tungsten solid solutions produced by cobalt substitutions. At about the same time in an

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excellent survey of this ternary system Köster and Tonn (3) described the temperature-composition limits of the several phases and in a second publication Köster (4) dealt with the hardening characteristics and magnetic properties.

ACKNOWLEDGMENTS

In expressing my appreciation of the honor conferred upon me as the twelfth Campbell Memorial Lecturer, I wish to share it among several men who have in one way or another contributed largely to the data upon which this discussion is built. Finley H. Ellinger of the Cleveland Wire Works staff is responsible for all dilatometric data and many of the lengthy treatments essential to the construction of the constitutional diagrams. His predecessor, Howard F. Graff, had earlier explored the ternary system sufficiently to recognize the true nature of some of the characteristic structures. A. B. Gladding with his customary skill prepared many indispensable X-ray photo-To L. W. Kempf and H. H. Hunsicker of the Aluminum Co. of America I am indebted for microcharacter hardness measurements and photomicrographs, while Dr. Kent R. Van Horn of the same laboratories co-operated in furnishing X-ray diffraction data. During the past six years several senior students in the department of Metallurgical Engineering at Case School of Applied Science have investigated in their thesis work certain aspects of this ternary system. This group consists of Francis P. Whalen, Harris P. Moyer, Frank Humberger, John W. Baer, Edward E. Stonebrook, Wm. Mogg and Richard Senz.

Throughout the course of these studies, Dr. Zay Jeffries, by his sustained interest and pertinent suggestions, has provided an ever welcome source of inspiration.

INTRODUCTION

The alloys under consideration today lie in the iron-rich portion of the ternary composition triangle (Fig. 1) and maybe considered as iron-tungsten alloys in which a part of the iron is replaced by cobalt. A brief review of the structures and hardening characteristics in this portion of the iron-tungsten system may at this point be included as a profitable introduction to the ternary alloys.

By the addition of tungsten to iron the A_4 point is lowered and the A_3 raised resulting in restriction of the gamma field. Between

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3.5 and 6 per cent tungsten both the gamma and alpha solid solutions exist over a range of temperature defined by the crescent shaped area in Fig. 2. If the tungsten content exceeds some 6 per cent only the alpha form of iron appears at any temperature below the freezing point. Alpha iron can hold in solid solution about 30 per cent of tungsten at 1500 degrees Cent. (2730 degrees Fahr.), but at 600 de-

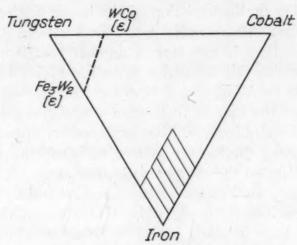


Fig. 1 — Ternary Composition Triangle, Quasi-binary Sections Indicated by Ruled Lines.

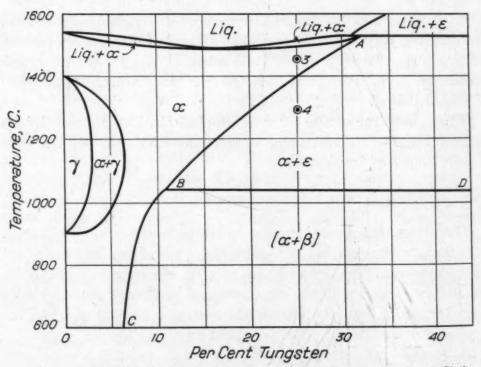


Fig. 2—Iron-rich Portion of Iron-tungsten Binary System. Numbered Circles Refer to Subsequent Microstructures.

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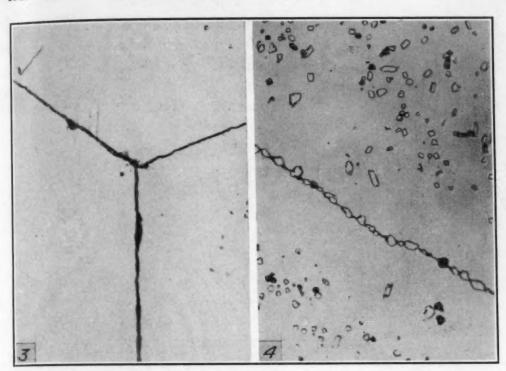


Fig. 3—Alpha Solid Solution. Iron 75 Per Cent, Tungsten 25 Per Cent. Quenched from 1450 Degrees Cent. as Shown by Location 3 in Fig. 2. × 200.

Fig. 4—Alpha + Epsilon. Iron 75 Per Cent, Tungsten 25 Per Cent. Quenched from 1300 Degrees Cent., Location 4 in Fig. 2. × 200.

grees Cent. (1110 degrees Fahr.) the solid solubility drops to 6 per cent.

In Fig. 3 appears the structure of the alpha solid solution, preserved by quenching from 1450 degrees Cent. (point 3 in Fig. 2) while upon slowly cooling to a temperature, 4, below the line AB, the tungsten separates from solid solution as relatively large particles of an intermediate phase corresponding to the formula Fe_3W_2 . This phase is designated as epsilon (ϵ) in the diagram and is visible in the microstructure of Fig. 4.

The hardening which results from quenching pure iron from a temperature within or above the gamma range is indicated in the lower diagram of Fig. 5. Tungsten in amounts up to 4.5 per cent accentuates this hardening action, which, however, vanishes quite abruptly as the tungsten content exceeds 6 per cent where, as will be observed, the two hardness curves merge. As will be shown presently this phenomenon bears directly upon the hardening characteristics of the ternary alloys and hence warrants here a limited survey of the attendant microstructures. When drastically quenched from or through the gamma range pure iron is characterized by relatively

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small grains with the serrated boundaries shown in Fig. 6. This is obviously the mark of arrested grain growth in the freshly formed alpha iron and offers striking contrast to the large grain of regular outline (Fig. 7) which results from cooling at a moderate rate through the same temperature range. In fact the former structure can be converted to the latter by reheating the quenched iron for

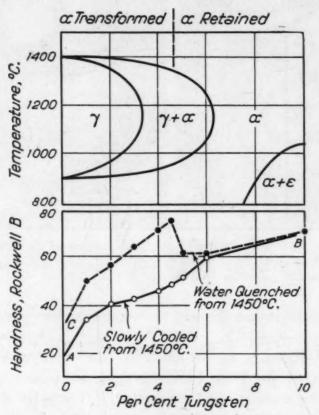


Fig. 5—Diagrams of Alpha-gamma Loop and Hardening Versus Cooling Rate of Alloys in this Composition Range.

degrees Fahr.). As might be expected such treatment likewise reduces the hardness of the quenched specimen to that of the slowly cooled.

This serrated form of grain is observed in alloys containing up to 4.5 per cent tungsten after quenching from a temperature within the gamma range or above the alpha-gamma range. In Fig. 8 are shown the structures to two alloys water quenched from 1400 degrees Cent. (2550 degrees Fahr.). Plainly a real grain refinement has occurred in the 3.5 per cent tungsten alloy at the left. On the other hand, the large alpha grains of the second structure indicate that in

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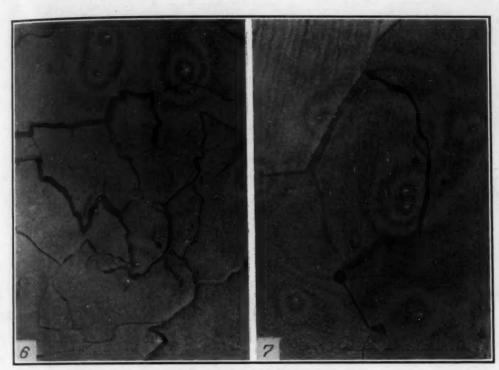
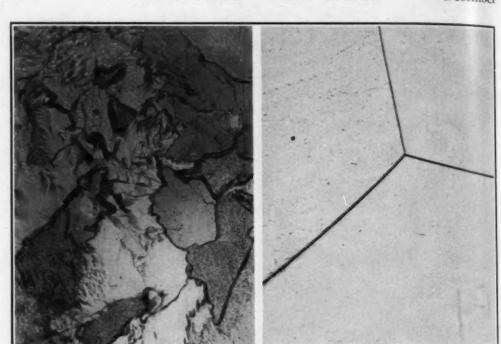


Fig. 6—Iron, Hydrogen Reduced from Precipitated Oxide, Melted and Frozen in Hydrogen. Quenched in Iced Brine from 1000 Degrees Cent. × 200.

Fig. 7—Iron, Hydrogen Reduced from Precipitated Oxide, Melted and Frozen in Hydrogen, Cooled from 1000 to 600 Degrees Cent. in 5 Hours. × 200.

the presence of 5 per cent tungsten no transformation will occur during such rapid cooling.

Obviously the presence of some 5 per cent tungsten is necessary to prevent a partial or entire lattice rearrangement in the high temperature alpha solid solution during cooling through the gamma range even though the cooling rate be extremely high. This alloy of 3.5 per cent tungsten when quenched from 1400 degrees Cent. (2550 degrees Fahr.) will apparently have undergone the double transformation throughout, but in Fig. 9 we observe that by heating for a few hours at a temperature within the alpha-gamma field, the two solid solutions may be segregated. After quenching from such a treatment the two solid solutions existing before the quench may be readily discerned in the microstructure. The lighter alpha phase of tungsten content "A" has remained unchanged during the quench, and the areas representing the gamma solid solution of composition "B" have of course transformed to alpha in cooling to room temperature. In this instance the overall tungsten content of 3.5 per cent was not sufficient to stabilize the high temperature alpha phase. By the segregating treatment, however, the tungsten was allowed to dis-



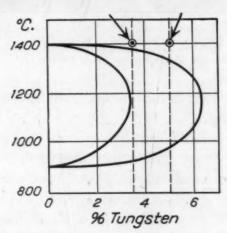


Fig. 8—Alloys of Iron Containing 3.5 and 5 Per Cent Tungsten Respectively. Quenched from 1400 Degrees Cent. × 200.

tribute itself between the two solid solutions and the alpha phase richer in tungsten was unaffected by the quench. Herein appears to lie the explanation for the sudden disappearance of the quench hardening effect as the tungsten content rises above 5 per cent in the binary iron-tungsten alloys.

As the tungsten content is increased from 6 per cent to 30 per cent, the hardness of the alpha solid solution rises correspondingly and a second glance at Fig. 5 shows moreover that the mixture of alpha and epsilon, in which the latter phase has been allowed to

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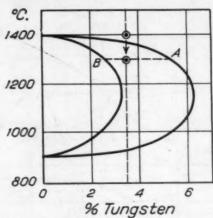


Fig. 9—Segregation of Gamma and Alpha Solid Solutions in 3.5 Per Cent Tungsten Alloy Slowly Cooled from 1400 to 1300 Degrees Cent. and Quenched. × 200.

precipitate as massive particles by furnace cooling, has substantially the same hardness as the alpha solid solution preserved by quenching.

When the coarse-grained solid solutions containing more than 6 per cent tungsten are quenched from the alpha range and reheated at 550 degrees Cent. (1020 degrees Fahr.) measurable precipitation hardening is observed after about 50 hours. At 600 degrees Cent. (1110 degrees Fahr.) the hardening occurs more rapidly and at 700 degrees Cent. (1290 degrees Fahr.) the maximum hardness is attained at the end of 50 hours as may be seen from the time-hardness

curves of Fig. 10. Hardened in this manner, such iron-tungsten alloys will obviously resist softening at temperatures of 600 or 700 degrees Cent. (1110 or 1290 degrees Fahr.) but are deficient in strength because of their large grain size.

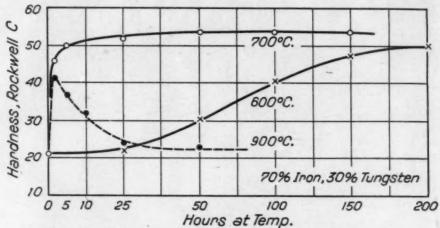


Fig. 10-Precipitation Hardening Curves of Alloy Containing 70 Per Cent Iron, 30 Per Cent Tungsten.

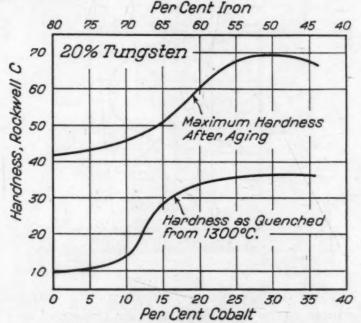


Fig. 11-Effect of Substituting Cobalt for Iron in 20 Per Cent Tungsten Alloy.

EFFECTS OF COBALT SUBSTITUTIONS

By substituting cobalt for a part of the iron in these alloys the solid solution is altered so as to become susceptible to grain refinement by rapid cooling from a temperature within the gamma range. Precipitation hardening may be induced within the fine-grained alpha

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by a subsequent treatment and the resulting alloy then combines the desirable high temperature hardness with the improved strength conferred by a small grain size of the alpha solid solution.

To illustrate the most obvious effects of cobalt substitutions we shall use as the base an alloy of 80 per cent iron and 20 per cent tungsten. Reasons for this selection will presently become apparent.

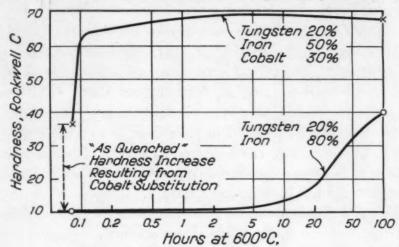


Fig. 12—Precipitation Hardening Curves of Binary Irontungsten Alloy and Ternary Iron-cobalt-tungsten Alloy.

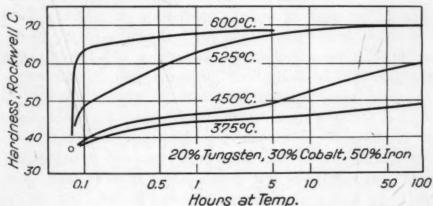


Fig. 13—Precipitation Hardening of Iron-cobalt-tungsten Alloy at Various Temperatures.

Two distinct changes in the hardness characteristics are immediately observed as the cobalt substitutions progress. The first of these changes, illustrated in Fig. 11, appears in the abrupt hardness rise as the cobalt content increases from 10 to 15 per cent. The lower curve, representing the hardness as quenched and the upper curve showing the maximum hardness attained by subsequent aging at an optimum temperature, both rise abruptly within this narrow range of cobalt content.

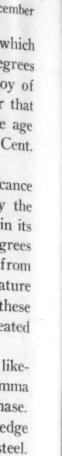
Equally surprising is the second effect shown in Fig. 12 which appears in the greatly increased rate of age hardening at 600 degrees Cent. (1110 degrees Fahr.) over that of the iron-tungsten alloy of equal tungsten concentration. In Fig. 13 we observe moreover that when the cobalt content is increased to 30 per cent, measurable age hardening occurs at a temperature as low as 375 degrees Cent. (705 degrees Fahr.).

Less apparent than these two effects but of equal significance is a third change in the hardening characteristics imparted by the presence of cobalt. The 30 per cent cobalt alloy in order to attain its maximum hardness by aging at 600 degrees Cent. (1110 degrees Fahr.) should previously have been cooled at a fairly rapid rate from about 1300 degrees Cent. (2370 degrees Fahr.) to some temperature below 450 degrees Cent. (840 degrees Fahr.). Only under these conditions will the rapid hardening occur when the alloy is reheated at 600 degrees Cent. (1110 degrees Fahr.).

Preliminary observations of these phenomena suggested the likelihood of reactions involving the lattice transformation of the gamma solid solution followed by the precipitation of a tungsten-rich phase. It should be recognized that this analysis is based upon our knowledge of austenite decomposition and the mechanism of hardening in steel.

In order to better understand the real nature of the hardening processes in these ternary alloys it will perhaps be helpful to examine hurriedly that portion of the iron-cobalt-tungsten constitutional diagram in which these compositions lie. The three limiting binary systems (5-6-7-8) are shown in the diagrams of Fig. 14. Although data are not available for positive confirmation, the probable form at the cobalt-rich end of the cobalt-iron system is indicated by the broken lines in the upper left-hand corner. Köster and Tonn, in their survey of this ternary system, showed sections parallel to the cobalt-iron side. In the present discussion, since the iron-tungsten system is to serve as a standard of reference, it seems desirable to illustrate the equilibrium structures by sections drawn parallel to the iron-tungsten side at intervals as indicated in Fig. 15.

At this time I wish to call to your attention certain important features in the cobalt-iron system which appear in the left-hand portion of the drawing. Observe, first, that with increasing cobalt the field of the delta (or upper alpha) solid solution grows narrower, being bounded on the lower side by the two-phase alpha-gamma field until at about 18 per cent cobalt the gamma form has completely



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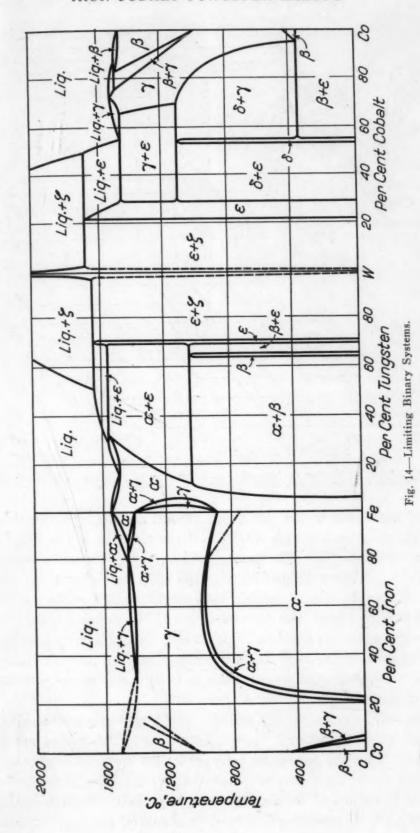
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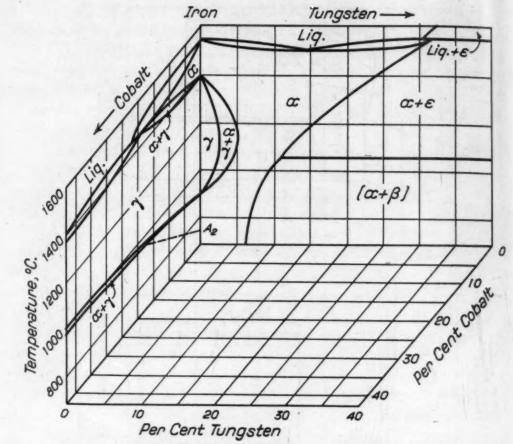


Fig. 15—Iron Corner of Iron-cobalt-tungsten System Showing Location of Sections Parallel to Iron-tungsten Side.

replaced the alpha in the solid. A second alpha-gamma field starts at the A₃ point in the iron corner and rises slowly in the shape of a narrow wedge forming the upper boundary of the low temperature alpha field. A dashed line indicates the shift to higher temperatures of the magnetic change which appears to merge with the alpha-gamma field at about 950 degrees Cent. (1740 degrees Fahr.) when the cobalt content approaches 15 per cent. Dilatometric and magnetic observations indicate that both the alpha-gamma and the magnetic changes are substantially reversible in cobalt-iron alloys within this range of composition.

The microstructure of the 30 per cent cobalt-iron alloy as quenched from the gamma state shown in Fig. 16 differs not at all from that found in pure iron after a similar treatment, although as a result of the cobalt substitution, the Brinell hardness in the quenched condition has doubled, increasing from 70 to 140. A striking change in the quenched structure, however, is observed when as little as 10

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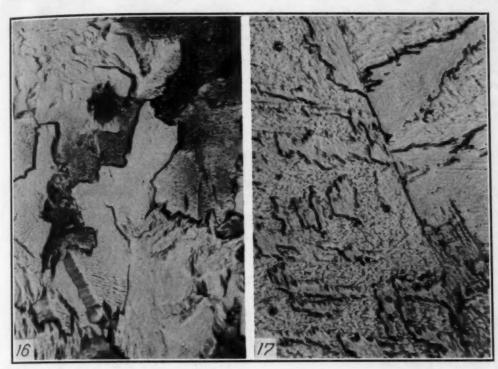


Fig. 16—Iron 70 Per Cent, Cobalt 30 Per Cent, Water-quenched from 1450 Degrees Cent. × 200.

Fig. 17—Iron 60 Per Cent, Cobalt 30 Per Cent, Tungsten 10 Per Cent, Water-quenched from 1450 Degrees Cent. × 200.

per cent of the iron is replaced by tungsten in the 30 per cent cobalt alloy. In this composition, a distinct Widmannstätten pattern appears as shown in Fig. 17, becoming more pronounced as the tungsten content approaches 20 per cent.

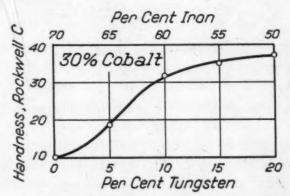


Fig. 18—Effect on Hardness Produced by Substituting Tungsten for Iron in Alloy of 30 Per Cent Cobalt as Quenched from 1300 Degrees Cent.

In Fig. 18 is shown the effect on hardness which results from replacing iron with tungsten in the 30 per cent cobalt alloy and we observe that the hardness after quenching from 1300 degrees Cent.

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	Table I									
	Nominal	Comp	osition	of	Alloys	Included	in This	Inves	tigation	
Alloy No.	Co	W	Mo		C	V	Ni	Cr	Mn	Fe
1		20%			0.01%					Bal
2		20%		-	0.01%	****	5%	5%	******	Bal
3		20%			0.01%			5%	*****	Bal
4	30%	18%	3		0.15%	2.5%			0.50%	Bal

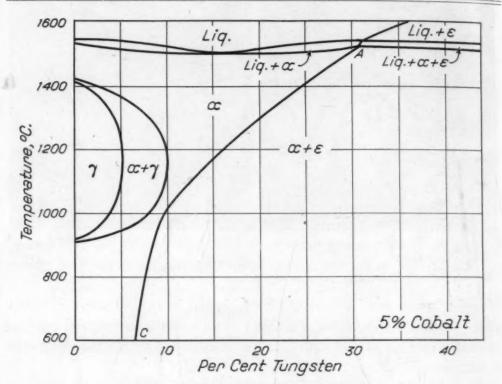


Fig. 19-Section at 5 Per Cent Cobalt.

(2370 degrees Fahr.) has attained a value of Rockwell C 37 as the tungsten content reaches 20 per cent.

Equilibrium Structures in the Ternary Alloys

For locating boundaries of the various phase fields about to be described, the data were obtained largely from the microstructures of alloys quenched from each one of a series of decreasing temperatures after holding at temperature for at least 20 hours. This treatment appeared in most cases sufficient to approximate structural equilibrium. In so far as possible the treatments were made cumulative in order to observe the alterations in a recognized structure brought about by a subsequent temperature change. In several instances specimens were quenched from a series of rising tempera-

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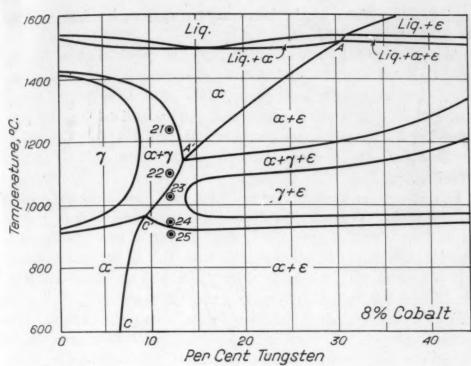


Fig. 20—Section at 8 Per Cent Cobalt. Numbered Circles Refer to Specimens in Subsequent Figures.

tures to more certainly establish the field boundary. The solidus and liquidus lines shown in the ternary sections have been taken largely from the diagrams of Köster and Tonn.

In Table I appears the nominal compositions of the four alloys to be discussed in this lecture. Except for alloy No. 4, all the material used for these determinations was hot-rolled from small ingots melted from carbon-free metals in a hydrogen atmosphere.

Starting from the iron-tungsten base, let us now make a short excursion into the region of increasing cobalt concentration. At our first stop, we shall view in Fig. 19 the section representing the 5 per cent cobalt alloy. Here both the gamma field and the gamma-alpha field are found to be broadening so that gamma iron can dissolve as much as 5 per cent tungsten at 1200 degrees Cent. (2190 degrees Fahr.) and some of the gamma exists together with the alpha solid solution at this temperature. No new structures appear as yet, but in order to preserve the alpha solid solution during a quench from 1400 degrees Cent. (2550 degrees Fahr.) at least 8 per cent cobalt must be present. The imminence of a new field is forecast by the impending intersection of the alpha-gamma crescent with AC, the boundary of the alpha field. For reasons to be forthcoming the in-

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At our next halt, where in Fig. 20 the cobalt content has increased to 8 per cent, we find distinct alterations in the map of the section. Two new fields now appear. One of these is defined by the presence of all three solid phases, alpha, gamma and epsilon, throughout considerable ranges of temperature and composition. This three-phase field incloses on three sides the second new field of gamma and epsilon.

In this section we find for the first time the structure formed when the alpha solid solution decomposes into gamma and epsilon upon cooling into the three-phase field. This is best shown by a comparison of four photomicrographs from the 12 per cent tungsten alloy. The structure shown in Fig. 21 displays a small patch of gamma formed from the alpha matrix after 20 hours at 1250 degrees Cent. (2280 degrees Fahr.) and transformed to alpha of course in the subsequent quench. The gamma phase increases in quantity upon lowering the temperature to 1100 degrees Cent. and results in the now familiar mixture of the two solid solutions in the alpha-gamma field, illustrated in Fig. 22. By cooling still further to 1050 degrees Cent. (1920 degrees Fahr.) and holding at this temperature for 20 hours we find, after quenching, that some of the alpha has decomposed into an intimate mixture of gamma and epsilon seen here as the darkly etched area in Fig. 23. This is replacing the smooth band of alpha in the center of the field, and results from the reaction which appears to have reached completion after 20 hours at 950 degrees Cent. (1740 degrees Fahr.), as may be judged from the structure of Fig. 24.

In this composition some alpha is always present and after cooling to 900 degrees Cent. (1650 degrees Fahr.) the gamma has been entirely converted to alpha within which are scattered the clusters of epsilon (Fig. 25) marking the position of its original precipitation at higher temperatures.

Moving now to the section at 10 per cent cobalt, we find in Fig. 26 the gamma field extending in the direction of higher tungsten concentrations. Here the alpha-gamma field appears in two distinct sections as does likewise the three-phase field. This location will furnish a composition which nicely illustrates some interesting details of structure. Let us first follow the changes which occur in a 9 per cent tungsten alloy between 1100 and 900 degrees Cent. As quenched

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Fig. 21—8 Per Cent Cobalt, 12 Per Cent Tungsten, 80 Per Cent Iron. Quenched After 20 Hours at 1250 Degrees Cent. γ Segregating from a. \times 200. Fig. 22—Same Alloy as in Fig. 21. Quenched After Holding 20 Hours Successively at 1250 and 1100 Degrees Cent. Alternate Bands of γ and a. \times 200. Fig. 23—Same Alloy as in Figs. 21 and 22. Quenched After Holding for 20 Hours Successively at 1250, 1100 and 1050 Degrees Cent. α Decomposing to Form γ + ϵ .

Fig. 24—Same Alloy as in Figs. 21, 22 and 23. Quenched After Holding for 20 Hours Successively at 1250, 1100, 1050 and 950 Degrees Cent. a Decomposition Nearly Complete. X 200.

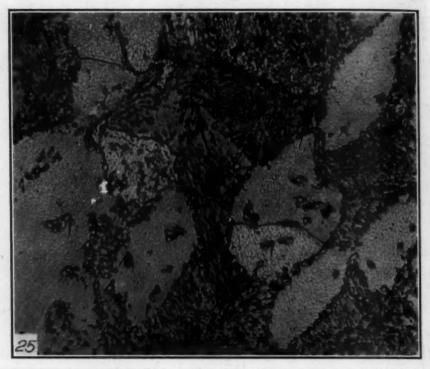


Fig. 25—8 Per Cent Cobalt, 12 Per Cent Tungsten, 80 Per Cent Iron. Held 20 Hours Successively at 1200, 1050, 950 and 900 Degrees Cent. Water Quenched. $a+\epsilon$. \times 200.

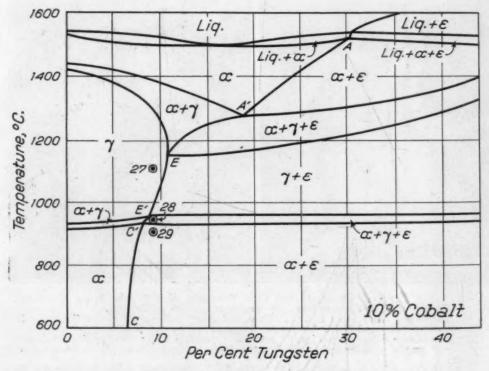


Fig. 26-Section at 10 Per Cent Cobalt.

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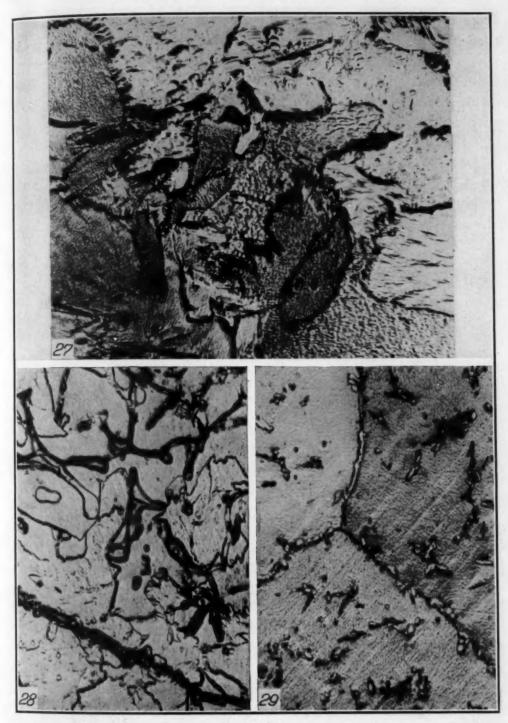


Fig. 27—10 Per Cent Cobalt, 9 Per Cent Tungsten, 81 Per Cent Iron. Quenched from 1100 Degrees Cent. \times 200. Fig. 28—Same Alloy as in Fig. 27. Slowly Cooled from 1100 to 950 Degrees Cent., Held 20 Hours at 950 Degrees Cent. and Quenched. $\alpha + \gamma + \epsilon$. \times 200. Fig. 29—Same Alloy as in Figs. 27 and 28. Held 20 Hours Successively at 1200, 950 and 900 Degrees Cent. and Quenched. $\alpha + \epsilon$. \times 200.

from 1100 degrees Cent. (2010 degrees Fahr.) the fragmented structure of Fig. 27 may be recognized as the result of the gamma to alpha transformation. This alloy was then cooled to 1000 degrees Cent. (1830 degrees Fahr.) where it remained for 20 hours, subsequently cooled again to 950 degrees Cent. (1740 degrees Fahr.), held at this temperature for 20 hours then quenched. After this treatment,

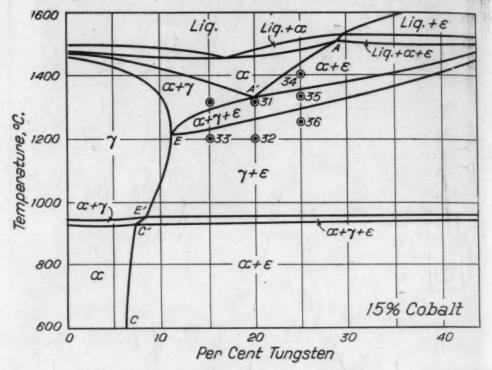


Fig. 30-Section at 15 Per Cent Cobalt.

we see from Fig. 28 that both alpha and gamma solid solution were present at 950 degrees Cent. (1740 degrees Fahr.) along with a scattering of the darkly outlined epsilon segregate which appears to have formed along the gamma grain boundaries during the treatment at 1000 degrees Cent. (1830 degrees Fahr.) in the gamma-epsilon field. Later, at 950 degrees Cent. (1740 degrees Fahr.) some of the gamma decomposed to form alpha and epsilon, the latter scattered as small globules throughout the newly formed alpha grains. Finally after cooling and holding at 900 degrees Cent. (1650 degrees Fahr.) for 20 hours the structure has been completely converted to the large grains of alpha and well defined crystals of epsilon seen in Fig. 29.

At 15 per cent cobalt (Fig. 30) we find the upper three-phase region growing narrower and shifting to higher temperatures at the expense of the overlying fields. Let us again observe the decomposiCooled to For

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Fig. 31—15 Per Cent Cobalt, 20 Per Cent Tungsten, 65 Per Cent Iron. Slowly Cooled from 1400 to 1300 Degrees Cent., Held 20 Hours and Quenched. a Decomposing to Form γ and ϵ . \times 200. Fig. 32—Same Alloy as in Fig. 31. Held Additional 20 Hours at 1200 Degrees Cent. and Quenched. $\gamma + \epsilon$. \times 200.

tion of the high temperature alpha solid solution in several compositions. In the case of the 20 per cent tungsten alloy/cooled slowly into the three-phase field through the temperature indicated by A', decomposition, starting from scattered centers, progresses outward into the lighter alpha matrix and over a period of hours at 1300 degrees Cent. (2370 degrees Fahr.) results in the formation shown in Fig. 31. Here the gamma and aggregate of epsilon form simultaneously and appear as islands within the lighter alpha matrix. As the temperature is further lowered to 1200 degrees Cent. (2190 degrees Fahr.) within the gamma-epsilon field, the alpha is entirely replaced by the products of its decomposition, seen in the photomicrograph of Fig. 32.

Broadly speaking, the composition A' is analogous to the eutectoid composition in a binary system since from the alpha solid solution of this one composition begins to form simultaneously two new solid phases upon cooling through the temperature A'. If the tungsten content lies on either side of A', however, some proeutectoid gamma or epsilon precipitates from alpha before the three-phase reaction sets in. In Fig. 33 is shown the 15 per cent tungsten alloy

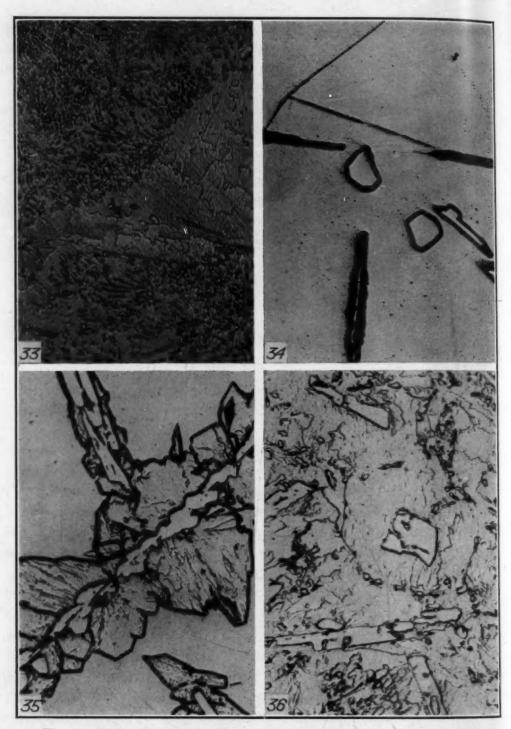


Fig. 33—15 Per Cent Cobalt, 15 Per Cent Tungsten, 70 Per Cent Iron. Slowly Cooled from 1400 to 1350 Degrees Cent. Held 20 Hours at 1350 and 1200 Degrees Cent., Quenched. Illustrates Formation of "Hypoeutectoid" γ at 1350 Degrees Cent. a Decomposed to γ + ε Upon Further Cooling to 1200 Degrees Cent. × 200.

Fig. 34—15 Per Cent Cobalt, 25 Per Cent Tungsten, 60 Per Cent Iron. Slowly Cooled from 1450 to 1400 Degrees Cent. Quenched After 20 Hours at 1400 Degrees Cent. Segregation of "Hypereutectoid" ε. × 200.

Fig. 35—Same Alloy as in Fig. 34. Cooled Further to 1325 Degrees Cent. Held 20 Hours and Quenched. Rim of γ Forming Around ε Segregate. × 200.

Fig. 36—Same Alloy as in Figs. 34 and 35. Held 20 Hours Successively at 1400, 1350, 1300 and 1250 Degrees Cent. Before Quenching, γ +ε. × 200.

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wherein the area of "proeutectoid" gamma was formed from alpha at 1350 degrees Cent. (2460 degrees Fahr.) and the remaining alpha decomposed at 1200 degrees Cent. (2190 degrees Fahr.) to form alpha and epsilon before quenching.

In the 25 per cent tungsten alloy we find that proeutectoid epsilon segregates as massive crystals from the alpha solid solution

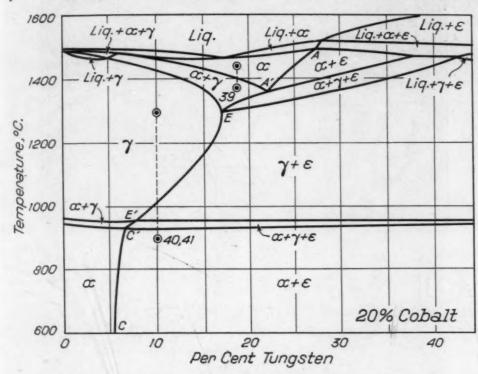


Fig. 37-Section at 20 Per Cent Cobalt.

when slowly cooled from 1450 to 1400 degrees Cent. (2640 to 2550 degrees Fahr.) (Fig. 34). Further cooling to 1325 degrees Cent. (2415 degrees Fahr.) results in the formation of gamma fringes usually growing outward from the epsilon segregate as seen in Fig. 35, while after a final period of 20 hours at 1250 degrees Cent. (2280 degrees Fahr.) the aggregate of gamma and epsilon appears as shown in the structure of Fig. 36.

Moving further into the realm of cobalt we begin to sense the ultimate fate of the "north-central provinces" in the 20 per cent cobalt section of Fig. 37 where, to use an expressive though vulgar idiom, they are obviously "on the way out." This location, however, furnishes something of interest in the 18 per cent tungsten alloy (Fig. 38) which is unable to retain the alpha when quenched from 1350 degrees Cent. (2460 degrees Fahr.) in the alpha-gamma field



Fig. 38—20 Per Cent Cobalt, 18 Per Cent Tungsten, 62 Per Cent Iron. 20 Hours at 1450 Degrees Cent., 20 Hours at 1350 Degrees Cent., Quenched. Dark Boundaries Indicate Existence of Both α and γ at 1350 Degrees Cent. α Has Undergone Transformation During Cooling in Quench Through γ Range. \times 100.

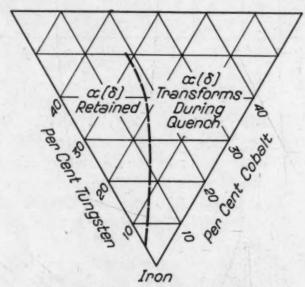


Fig. 39—Diagram Representing Relation Between Cobalt Content and Tungsten Content Necessary to Preserve α (or δ) During Quench.

and only by the distinct phase boundaries testifies to the existence of both the alpha and the gamma solid solutions at that temperature. In the iron-tungsten binary system, as you may recall, the presence December

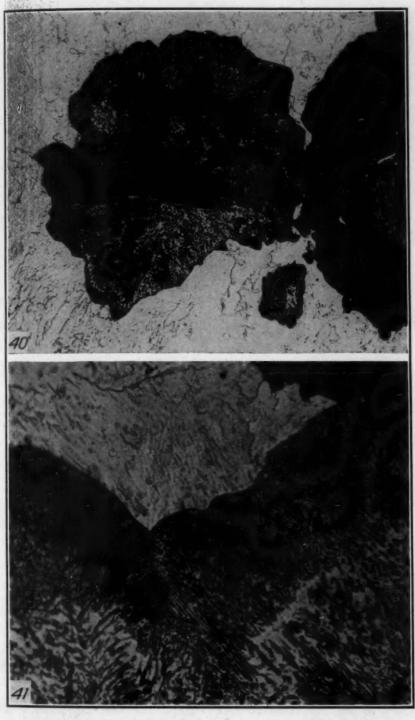


Fig. 40—20 Per Cent Cobalt, 10 Per Cent Tungsten, 70 Per Cent Iron. Held 20 Hours Successively at 1300, 1100, 1000, 950 and 900 Degrees Cent., Quenched. Shows γ Decomposing Into α + ε at 900 Degrees Cent.

Fig. 41—Same Specimen as in Fig. 40. × 500.

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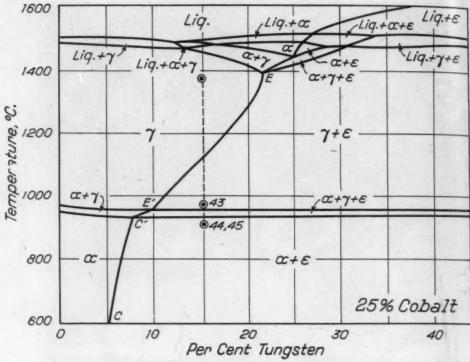


Fig. 42-Section at 25 Per Cent Cobalt.

of some 5 per cent tungsten is necessary to prevent the transformation of the alpha (or delta) solid solution in quenching from temperatures above the gamma range. With the introduction of cobalt, however, the tungsten requirement is increased and the limiting proportions of cobalt and tungsten necessary to retain the high temperature alpha form are approximated in Fig. 39 which illustrates a factor that is of necessity excluded from a diagram of equilibrium structures.

A formation of deeper significance (Fig. 40) is contributed by the 10 per cent tungsten alloy, which, upon cooling from the gamma range to a temperature just below the lower three-phase field, yields after 20 hours this pattern suggesting at once the familiar "rosette" of pearlite. The truly lamellar structure resolved at higher magnifications (Fig. 41) strengthens the analogy which appears to link this case with the isothermal decomposition of austenite at a temperature not too far below the critical. To further extend the comparison at the expense of precision, the point E' in this section may be said to parallel the eutectoid composition in the iron-carbon diagram.

We shall pause at the 25 per cent cobalt section (Fig. 42) only for a last glance at the high temperature fields of the alpha phase and to observe again the decomposition of gamma at 900 degrees

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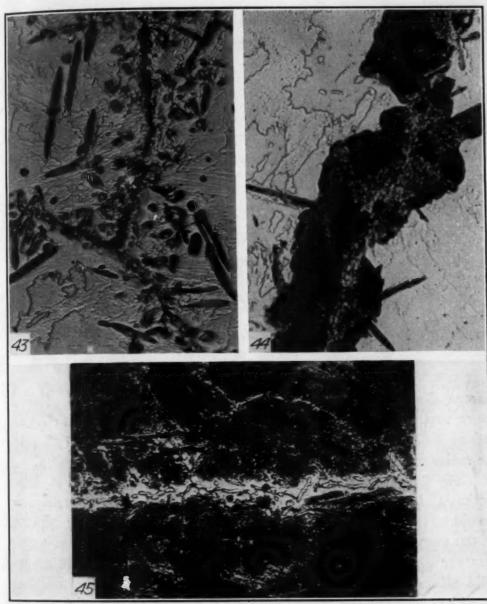


Fig. 43—25 Per Cent Cobalt, 15 Per Cent Tungsten, 60 Per Cent Iron. Cooled from 1400 Degrees Cent. to 950 Degrees Cent. in 80 Hours and Quenched. Segregate of ϵ in γ . \times 200. Fig. 44—Same Alloy as in Fig. 43, Cooled Further to 900 Degrees Cent. Held 20 Hours and Quenched. Beginning of γ Decomposition at 900 Degrees Cent. \times 200. Fig. 45—Same Alloy and Treatment as in Fig. 44, But Held at 900 Degrees Cent. for 40 Hours Before Quenching. The reaction $\gamma = \alpha + \epsilon$ Nearing Completion. \times 200.

Cent. (1650 degrees Fahr.). This alloy bearing 15 per cent tungsten was cooled from 1400 to 950 degrees Cent. (2550 to 1740 degrees Fahr.) over a period of 80 hours. As the temperature dropped below the line EE' epsilon precipitated from gamma, first at the grain boundaries, then within the grains, as may be observed in Fig. 43. Upon cooling further to 900 degrees Cent. (1650 degrees Fahr.)



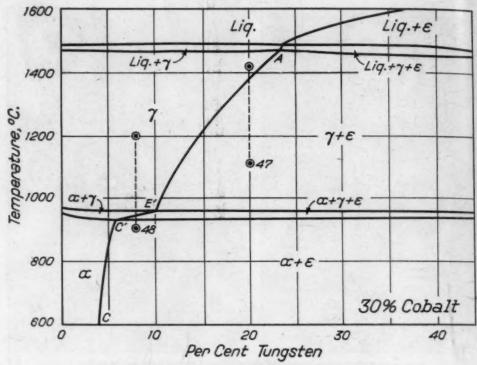


Fig. 46-Section at 30 Per Cent Cobalt.

gamma decomposition began at the grain boundaries and after 20 hours progressed outward to the extent shown in Fig. 44. At the end of 40 hours, the reaction had neared completion and (Fig. 45) the entire field has been darkened rapidly by the etchant.

The end of our excursion is reached at the 30 per cent cobalt section (Fig. 46) where we find the higher temperature ranges quite devoid of the alpha solid solution, this phase having been replaced by the gamma form. Here the 20 per cent tungsten alloy at a temperature above the line AE' consists of a single phase, the gamma solid solution. In this section is found the composition earlier selected to represent the typical alloy of the group, for, as the diagram illustrates, a tungsten concentration of 20 per cent approximates saturation in the gamma solid solution bearing 30 per cent cobalt. In cooling to room temperature, even by drastic quenching, the gamma is so thoroughly converted to the low temperature alpha form that the X-ray is unable to detect any residue. As previously mentioned this lattice change imparts to the final alpha structure the Widmannstätten pattern shown earlier (in Fig. 17) and results in a considerable increase in hardness over that of the cobalt-free alloy of like tungsten content. With very slow cooling from 1400 to 1100 degrees Cent. (2550 to 2010 degrees Fahr.) the gamma grain boundaries are from

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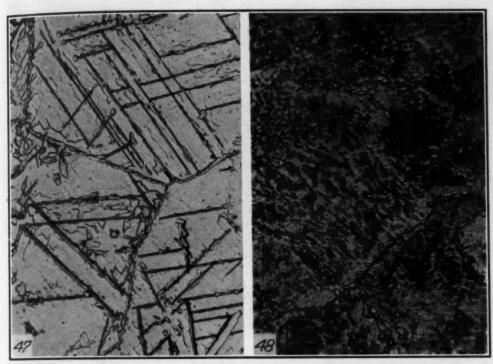


Fig. 47—30 Per Cent Cobalt, 20 Per Cent Tungsten, 50 Per Cent Iron. Cooled from 1400 to 1100 Degrees Cent, in 80 Hours. Quenched. $\gamma + \epsilon$. \times 200. Fig. 48—30 Per Cent Cobalt, 8 Per Cent Tungsten, 62 Per Cent Iron. Cooled from 1200 to 900 Degrees Cent. in 80 Hours. Quenched. $\alpha + \epsilon$. \times 200.

outlined by a thick network of epsilon precipitate and within the gamma grains this phase segregates as long needles forming again this well defined Widmannstätten pattern (Fig. 47).

It will be observed that since the first appearance of the lower three-phase field in the 8 per cent cobalt section its position has remained substantially fixed although the structures developed within this field are modified somewhat by changes in concentration of cobalt and tungsten. In the presence of 30 per cent cobalt an alloy containing as little as 8 per cent tungsten after very slowly cooling through 900 degrees Cent. (1650 degrees Fahr.) bears the usual evidence of this three-phase reaction in the highly dispersed state of the epsilon precipitate formed during the gamma decomposition and shown in Fig. 48.

In the sections viewed thus far, the solid three-phase field has appeared as though existing in two quite separate divisions. The true nature of this three-phase space, or "Dreiphasenraum" as the Germans have it, may perhaps be better understood from the vertical section in Fig. 49. This is erected parallel to the iron-cobalt side of the ternary diagram at the 20 per cent tungsten composition, and

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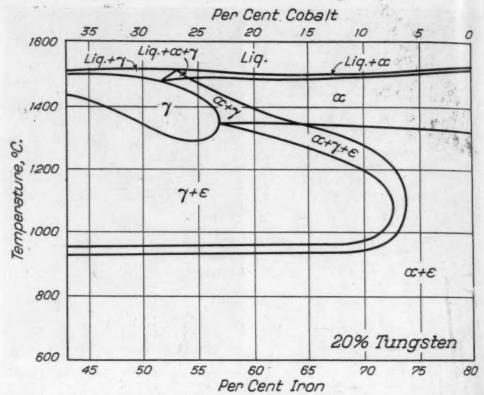


Fig. 49-Section Parallel to Iron-cobalt Side at 20 Per Cent Tungsten.

demonstrates that in alloys of this tungsten content the three-phase field will be discontinuous only within a limited range of cobalt concentrations. In alloy No. 1, bearing 30 per cent cobalt, this field is encountered only at temperatures between 925 and 950 degrees Cent. (1700 and 1740 degrees Fahr.) approximately.

Thus far, in order to readily identify the existing phases, we have been concerned with structures formed under conditions approaching equilibrium, i.e., resulting from extremely low rates of cooling. Some unusual effects may be produced, however, by drastically altering the conditions of equilibrium and following the progress of the changes in structure over a period of time. This can be most readily accomplished by rapidly transferring the alloy from one temperature at which it has attained structural equilibrium to another temperature differing from the first by several hundred degrees.

Returning for a moment to the 15 per cent cobalt section, let us watch the progress of alpha decomposition in a 20 per cent tungsten alloy which after heating at 1450 degrees Cent. (2640 degrees Fahr.) for 2 hours was transferred to a furnace at 1100 degrees Cent. (2010 degrees Fahr.) and held for periods of 10 and 60 minutes before

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Fig. 50—15 Per Cent Cobalt, 20 Per Cent Tungsten, 65 Per Cent Iron. Transferred from 1450 to 1100 Degrees Cent. and Held for 10 Minutes Before Quenching. \times 200. Fig. 51—Same Alloy and Treatment as in Fig. 50 But Held at 1100 Degrees Cent. for One Hour Before Quenching. \times 200. Fig. 52—15 Per Cent Cobalt, 20 Per Cent Tungsten, 65 Per Cent Iron. Held for 20 Hours Successively at 1350, 1300, and 1250 Degrees Cent. Quenched. γ + ϵ . \times 500.

quenching in water. The structures developed over these time intervals are better illustrated than described and appear in Figs. 50 and 51. This same alloy after slowly cooling through the same temperature range displays in Fig. 52 a quite dissimilar structure. Or again in the same 15 per cent cobalt group the "hypereutectoid" alloy of 25 per cent tungsten was transferred from 1450 to 1100 degrees Cent. (2640 to 2010 degrees Fahr.) and held at 1100 degrees Cent. for periods of 1 minute and five minutes before water quenching. In Figs. 53 and 54 are seen the structures resulting from the decomposition of alpha at 1100 degrees Cent. (2010 degrees Fahr.) to form gamma and epsilon over such short intervals of time. One of the symmetrical patterns in epsilon needles developing at the end of 5 minutes is shown in Fig. 55 at a higher magnification.

NATURE OF THE TUNGSTEN RICH PHASE (EPSILON)

In the binary iron-tungsten system an intermediate phase occurs which approximates the composition represented by the formula Fe₃W₂, as indicated in the composition triangle of Fig. 1, together with a corresponding intermediate phase represented by CoW which occurs in the cobalt-tungsten system. Köster and Tonn, on the basis of etching reactions observed in several compositions lying between Fe₃W₂ and CoW, concluded that iron and cobalt replaced one another in all proportions without the formation of a new phase. The series of X-ray photograms shown in Fig. 56 confirms this conclusion. These photograms were prepared from alloys formed by heating the pressed mixtures of metal powders for 50 hours at a temperature just below the solidus.

The crystal form of Fe₃W₂ as determined by Arnfelt (9) is trigonal. Hence we may conclude that Fe₃W₂ and CoW represent the extremities of an isomorphous series crystallizing in the trigonal system and changing but slightly in axial ratio and parameter as cobalt replaces iron.

The ternary iron alloy containing 30 per cent cobalt and tungsten was treated to precipitate from the gamma solid solution a considerable quantity of coarse epsilon by cooling slowly from 1450 to 1200 degrees Cent. (2640 to 2190 degrees Fahr.). From precipitate isolated by electrolytic solution a photogram was obtained which appears at the bottom in Fig. 56 and is readily identified as belonging to the series Fe₃W₂-CoW.

A second intermediate phase has been identified in both the iron-

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tungsten and cobalt-tungsten systems which forms very slowly by a peritectoid reaction at 1000 to 1100 degrees Cent. (1830 to 2010 degrees Fahr.). These low temperature phases which differ in crystal form might be expected to introduce a third phase in the ternary system by some type of reaction at a relatively low temperature. Attempts to isolate the tungsten-rich precipitate formed below 900 degrees Cent. (1650 degrees Fahr.) were unsuccessful due to its highly dispersed state. Photograms from block specimens containing this precipitate yielded no positive information as to its nature. Consequently, only one tungsten-rich phase has been included in the ternary sections under discussion and a fuller knowledge of the low temperature fields must await further investigation.

VOLUME CHANGES

Earlier mention has been made of the nearly reversible nature of the gamma-alpha change in iron-cobalt alloys. The 30 per cent cobalt composition will serve to illustrate this characteristic and from Fig. 57 it is apparent from the dilatometer curves that the volume change begins at about 900 degrees Cent. (1650 degrees Fahr.) upon heating as well as upon cooling. The onset of this transformation appears to be independent of the temperature from which cooling starts. This observation is worthy of especial emphasis in view of certain phenomena to be presently described.

Substitution of tungsten for iron in the 30 per cent cobalt alloy causes the alpha-gamma change to occur at gradually lowering temperatures upon heating. In Fig. 58 the contraction accompanying this change is shown in the dilatometer curve "A" for the 30 per cent cobalt, 20 per cent tungsten alloy, where the decrease in length sets in at about 960 degrees Cent. (1760 degrees Fahr.) attaining completion at 980 degrees Cent. (1795 degrees Fahr.). After holding this alloy at 1050 degrees Cent. (1920 degrees Fahr.) for several hours, then cooling in the dilatometer, the course of the curve "B" shows the gamma-alpha expansion, beginning at about 930 degrees Cent. (1705 degrees Fahr.) but proceeding very slowly until at 900 degrees Cent. (1650 degrees Fahr.) the rate of change rises suddenly and the expansion is complete at 870 degrees Cent. (1600 degrees Fahr.).

Let us compare with this the third curve, "C", representing the course of the gamma-alpha change in cooling from 1300 degrees Cent. (2370 degrees Fahr.). Here the expansion takes place in two distinct

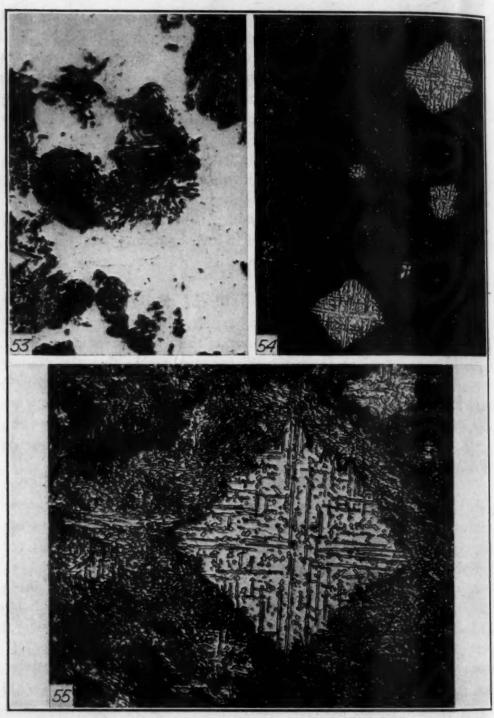


Fig. 53—15 Per Cent Cobalt, 25 Per Cent Tungsten, 60 Per Cent Iron: Transferred from 1450 to 1100 Degrees Cent. and Held for 1 Minute Before Quenching. × 500.

Fig. 54—Same Alloy and Treatment as in Fig. 53 But Held 5 Minutes at 1100 Degrees Cent. Before Quenching. × 100.

Fig. 55—Same Specimen as in Fig. 54. × 500.

steps, the first a slight indication at about 900 degrees Cent. (1650 degrees Fahr.); the second and major length increase is delayed until

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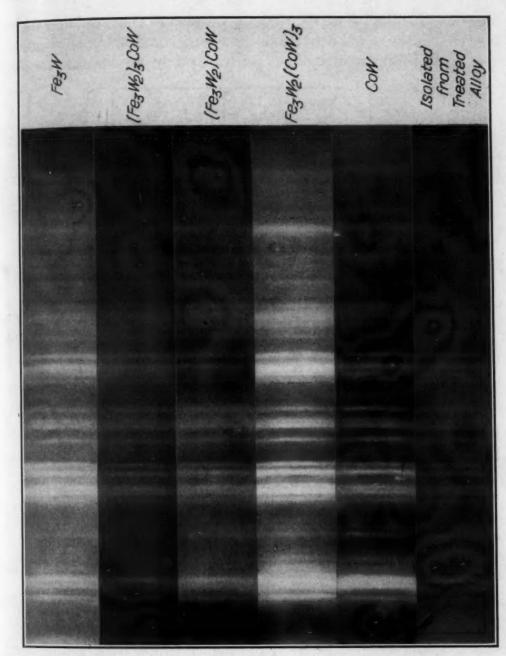


Fig. 56—Photograms of Isomorphous Series Fe₃W₂-CoW, Identified as ε Phase.

the temperature has fallen to about 480 degrees Cent. (895 degrees Fahr.). The rates of heating and cooling between 800 and 1000 degrees Cent. (1470 and 1830 degrees Fahr.) approximated 1 degree Cent. per minute in all cases.

Since the curves B and C in Fig. 58 resulted from nearly identical cooling rates it becomes obvious that the temperature of the gamma-alpha transformation is closely related to the temperature of

previous treatment, i. e., the solution treatment. The cooling rate is a second factor which determines the temperature of transformation. With sufficiently rapid cooling, we may safely assume that no ex-

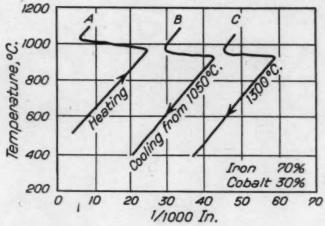


Fig. 57-Dilatometer Curves of Iron-cobalt Alloy.

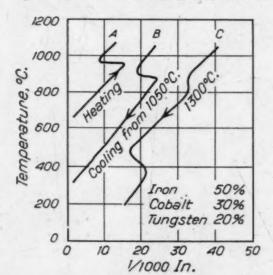


Fig. 58-Dilatometer Curves of Iron-cobalt-tungsten Alloy.

pansion would have occurred at the higher temperature in the case of curve C. Such data are reminiscent of the time-temperature curves obtained by H. C. H. Carpenter (10) from a study of the critical ranges of certain high speed steels. This work was performed more than thirty years ago at the National Physical Laboratory in England. Summarizing his findings Carpenter commented in part as follows: "The hardening of rapid tool steels at the present time appears to involve the widening, splitting or lowering of the critical ranges by the special alloy element (added to the steel)." Gill and

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Bowman (11) in 1921 again reported this form of cooling curve in high speed steels and today all students of metallurgy are in some degree familiar with the phenomenon of the "split transformation."

ISOTHERMAL DECOMPOSITION OF THE GAMMA SOLID SOLUTION AT SUBCRITICAL TEMPERATURES

Following the trail blazed by Davenport and Bain (12) we may arrive at a fuller understanding of the time-temperature relations governing the gamma to alpha transformation by causing this reaction to proceed isothermally and observing the accompanying volume

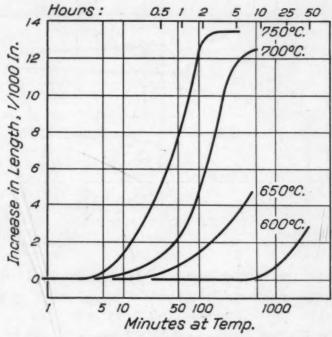


Fig. 59—Subcritical Isothermal Expansion Curves of Alloy No. 1 Following Solution Treatment at 1300 Degrees Cent.

change. Thus by rapidly transferring the alloy specimen from some predetermined temperature within the gamma range to the dilatometer maintained at the desired constant temperature, expansion is observed to begin after a certain lapse of time.

The time required for the onset of the transformation in a given alloy depends upon, first, the temperature of initial heating and second, the temperature of the specimen within the dilatometer. As examples of isothermal transformation at four temperatures we see in Fig. 59 the dilatometric curves for alloy No. 1 which after heating for 1 hour at 1300 degrees Cent. (2370 degrees Fahr.) was quickly transferred

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to the dilatometer at the designated temperatures. Plainly the beginning of expansion is delayed more and more as the temperature of decomposition is lowered.

An alternate method of determining the onset of the gamma transformation makes use of the appearance of ferro-magnetism in the alloy attending the formation of the alpha solid solution. While

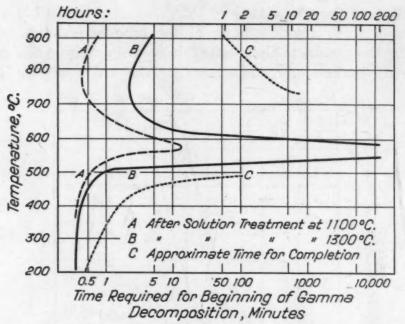


Fig. 60-Time-temperature (S) Curves for Gamma Decomposition.

less precise than the former method, this can be used to advantage with simpler equipment and is especially useful at elevated temperatures.

We see in Fig. 60 the so-called "S" curve for alloy No. 1 following a solution treatment of 1 hour at 1300 degrees Cent. (2370 degrees Fahr.). The solid line, B, indicates the time required for the onset of gamma decomposition. For the completion of this reaction, the required time may be closely estimated at temperatures above 600 degrees Cent. (1110 degrees Fahr.) from the dilatometric data as well as from the microstructure and is indicated by curve "C" in this diagram. Below 600 degrees Cent. (1110 degrees Fahr.) the completion of the transformation is less sharply marked. It may be approximated, as will be later explained, from the extent to which the alloy develops its full hardness upon directly transferring to 600 degrees Cent. (1110 degrees Fahr.) from the temperature of isothermal transformation.

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If the solution treatment is carried on at a temperature lower than 1300 degrees Cent. (2370 degrees Fahr.) the resulting "S" curve for this alloy is shifted toward the left; solution temperatures higher than 1300 degrees Cent. (2370 degrees Fahr.) move it toward the right. Curve "A" in Fig. 60 approximates the time-temperature relations following solution treatment at 1100 degrees Cent. (2010 degrees Fahr.) and illustrates that less time will elapse before the gamma transformation sets in.

In order to follow the changes in structure and hardness which accompany the isothermal transformations we have only to quench small blocks of the alloy from a selected temperature within the gamma range using as a quenchant some liquid medium maintained at the desired temperature of transformation between 900 and 100 degrees Cent. (1650 and 217 degrees Fahr.). By withdrawing specimens from the bath at intervals and water quenching we may follow the progress of the reaction from the accompanying microstructures. We shall learn, incidentally, something of the hardening effects attending decomposition throughout this range of temperature. It will be recalled that we are now concerned with the carbon-free iron base alloy containing 30 per cent cobalt and 20 per cent tungsten, which is designated as alloy No. 1. As quenched from 1300 degrees Cent. (2370 degrees Fahr.) into molten salt at 900 degrees Cent. (1650 degrees Fahr.), held at that temperature for 10 minutes then water quenched, the resulting structure of Fig. 61 reveals the progress of the decomposition. The light areas in the photomicrograph represent fields of gamma which were still intact after 10 minutes at 900 degrees Cent. (1650 degrees Fahr.) but which of course changed to alpha during the final quench to room temperature. After an hour at 900 degrees Cent. (1650 degrees Fahr.), the decomposition is nearly complete as shown by the etched microsection. In the few gamma areas remaining after one hour at 900 degrees Cent. (1650 degrees Fahr.) we see in Fig. 62 a scattering of epsilon particles which have attained visible dimensions. It is evident, therefore, that in the decomposition of gamma at 900 degrees Cent. (1650 degrees Fahr.), the allotropic transformation is preceded by precipitation of the tungsten-rich phase. The lamellar nature of the decomposition products is revealed in Fig. 63 at a magnification of 2000 diameters, though the details are much less distinct than those of the similar structure formed in the alloy of 20 per cent cobalt and 10 per cent tungsten (Fig. 40).

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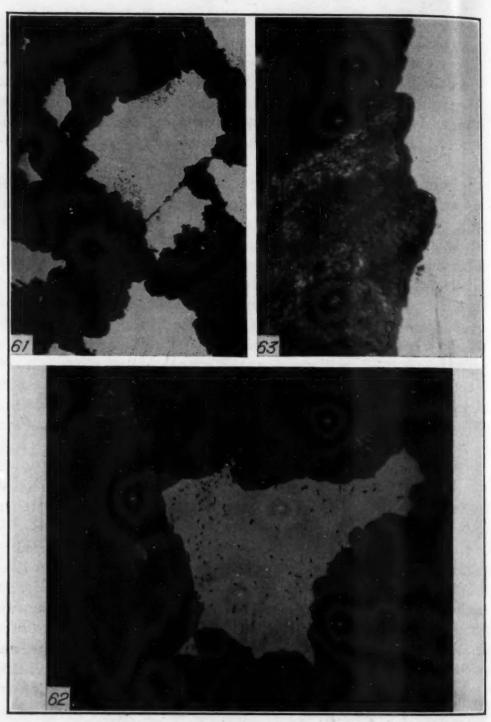


Fig. 61—30 Per Cent Cobalt, 20 Per Cent Tungsten, 50 Per Cent Iron. Quenched from 1300 to 900 Degrees Cent., Held 10 Minutes and Water Quenched. × 100.

Fig. 62—Same Alloy as in Fig. 61. Quenched from 1300 to 900 Degrees Cent. Held 1 Hour and Water Quenched. Visible Precipitate of ε Within Residual γ Field. × 1000.

Fig. 63—Same Specimen as in Fig. 61. × 2000.

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In curve A of Fig. 64 is shown the hardness change accompanying decomposition at 900 degrees Cent. (1650 degrees Fahr.), and in the initial horizontal portion of the curve may be recognized the short time interval preceding the start of the reaction. Once under way, however, the hardening cycle rapidly reaches a maximum and falls away to approach the original hardness after some 20 hours. Prior to the completion of gamma decomposition, which from the microstructure requires about one hour, the measured hardness after quenching to room temperature is the resultant of two structures, the alpha solid solution which transformed only during the final cooling

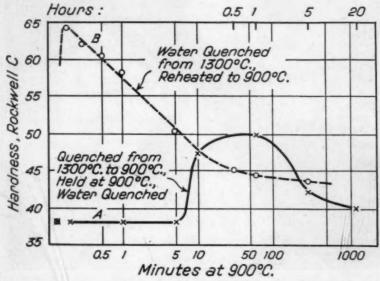


Fig. 64-Hardening at 900 Degrees Cent. in Alloy No. 1.

and the duplex structure of alpha and epsilon formed from gamma at 900 degrees Cent. (1650 degrees Fahr.).

Curve B in Fig. 64 illustrates the course of the hardening cycle observed in the alloy after first water quenching from 1300 degrees Cent. (2370 degrees Fahr.) then reheating at 900 degrees Cent. (1650 degrees Fahr.) in a salt bath. In this case we have forced the gamma-alpha transformation to take place at room temperature and then by reheating to 900 degrees Cent. (1650 degrees Fahr.) have allowed the precipitation of epsilon to proceed at that temperature within the fine-grained alpha matrix. This process goes on so rapidly that after immersion for 10 seconds in a salt bath at 900 degrees Cent. (1650 degrees Fahr.) the alloy has increased in hardness from C 38 to C 64, but as might be expected overages rapidly.

Upon quenching from 1300 to 750 degrees Cent. (2370 to 1380

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degrees Fahr.) the reaction begins with less delay than at 900 degrees Cent. (1650 degrees Fahr.) but moves more slowly to completion. As may be seen from Fig. 65 the product of gamma decomposition at 750 degrees Cent. (1380 degrees Fahr.) fails to display the lamellar appearance observed after the 900 degrees Cent. (1650 degrees Fahr.) reaction, but assumes now a needle-like or feathery

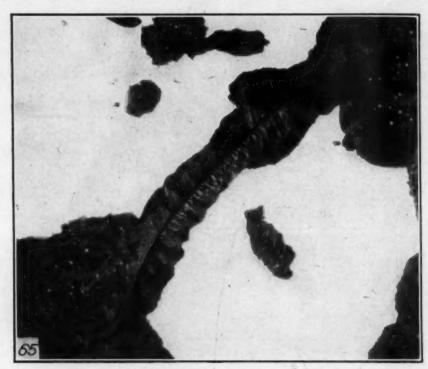


Fig. 65—30 Per Cent Cobalt, 20 Per Cent Tungsten, 50 Per Cent Iron. Quenched from 1300 to 750 Degrees Cent., Held 1 Hour and Water Quenched. × 500.

form extending outward from a gamma grain boundary or from centers within the grain, while at higher magnification evidence of a lamellar nature is still visible. Measurable hardening is observed after five minutes at 750 degrees Cent., but as shown in Fig. 66 the cycle is completed much more slowly than at 950 degrees Cent. (1740 degrees Fahr.).

As the temperature of decomposition is lowered to 600 degrees Cent. (1110 degrees Fahr.) structural evidence of the reaction is first encountered after an interval of 5 hours. The progress at the end of 25 hours is seen in Fig. 67, a structure which corresponds to a hardness of C46. The gamma decomposition obviously begins at the grain boundaries and likewise about the particles of epsilon which were not dissolved by the treatment at 1300 degrees Cent. (2370

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degrees Fahr.). After 75 hours at 600 degrees Cent. (1110 degrees Fahr.) decomposition has progressed to the stage pictured in Fig. 68, the hardness meanwhile having reached C60.

Curve A in Fig. 69 represents the course of hardening which accompanies isothermal decomposition at 600 degrees Cent. (1110 degrees Fahr.). No hardening is observed until some 5 hours have

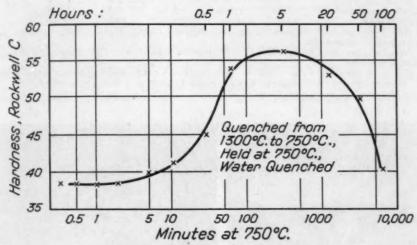


Fig. 66-Hardening at 750 Degrees Cent.

elapsed, although after 100 hours it is evident that the hardness is approaching that developed by the 600 degrees Cent. (1110 degrees Fahr.) aging of the water quenched alloy. In this figure such a "normal" hardening curve, B, is included for a twofold purpose. That by two such devious courses the hardness of a material should attain values substantially identical is in itself worthy of observation. More important, however, is the fact, demonstrated by curve B, that in a fine-grained alpha matrix the precipitation and attendant hardening proceed at an extremely rapid rate when the alloy is reheated to 600 degrees Cent. (1110 degrees Fahr.) and has attained a maximum of C 66-67 Rockwell within an hour's time. The significance of this phenomenon will become apparent as we consider the progress of isothermal decomposition and hardening at temperatures below 600 degrees Cent. (1110 degrees Fahr.). If the alloy be quenched from 1300 to 550 degrees Cent. (2370 to 1020 degrees Fahr.) and held at the latter temperature, no measurable hardening is observed within the first one hundred hours. This reluctance to harden is to be expected from the shape of the "S" curve, which indicates that at 550 degrees Cent. (1020 degrees Fahr.) the gamma transformation does not begin within this interval of time.

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Table II
Micro Hardness of Needles and Matrix in Alloy
No. 1 After Treatment Described

and the second	Specimen No. 1 1 Hr. at 500°C.			Specimen No. 2 50 Hrs. at 500°C.		Specimen No. 3 5 Hrs. at 500°C. 1 Hr. at 600°C.	
	Matrix	Needles	Matrix	Needles	Matrix	Needles	
Avg. width of							
scratch in microns	2.74	2.34	2.34	1.25	2.60	1.4	
		1830		6430	1480	5100	
Specimen No. 1—Quenched Cent. and water quenche	d.						
Specimen No. 2—Quenched and water quenched.							
Specimen No. 3—Quenched Cent. Transferred to 60	from 1300 00 degrees	to 500 de Cent. He	egrees Cent.	Held 5 d water	hours at quenched.	500 degre	

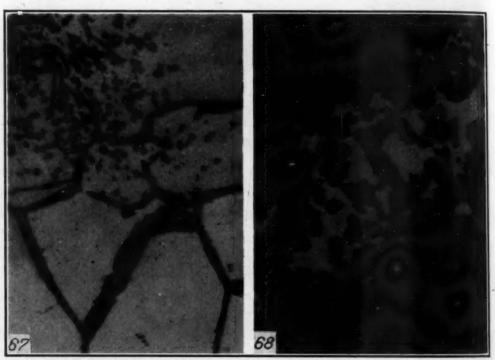


Fig. 67—Alloy No. 1. Quenched from 1300 to 600 Degrees Cent., Held for 25 Hours and Water Quenched. × 500.

Fig. 68—Alloy No. 1. Quenched from 1300 to 600 Degrees Cent., Held for 75 Hours and Water Quenched. × 500.

At 500 degrees Cent. (930 degrees Fahr.) the formation of alpha is underway again at the end of a few minutes and in alloy specimens, water quenched from 500 degrees Cent. (930 degrees Fahr.) we find the presence of alpha revealed in these scattered areas of acicular pattern (Fig. 70) outlined only by extended etching and suggesting in appearance the familiar martensite in a freshly quenched steel. Even though these needles at first etch with difficulty they are shown

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to be substantially harder than the surrounding matrix by the microcharacter test as seen in the photomicrograph of Fig. 71 and in Table II. With prolonged heating at 500 degrees Cent. (930 degrees Fahr.) these areas multiply and some are darkened rapidly by the etchant, as in Fig. 72, indicating the presence of a highly dispersed precipitate within the alpha needles. The microcharacter in fact

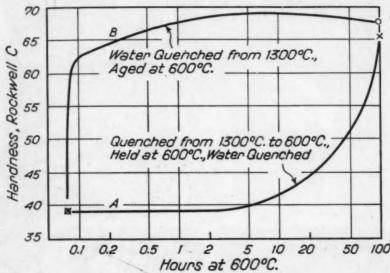


Fig. 69-Hardening at 600 Degrees Cent.

shows that a remarkable hardness increase has occurred within the needles, the micro hardness number "K" having increased from 1830 to 6430 as the time at 500 degrees Cent. (930 degrees Fahr.) is prolonged from 1 hour to 50 hours. It appears moreover (from Table II) that during this time interval substantial hardening has taken place within the matrix. The considerable increase in Rockwell C hardness which attends this precipitation at 500 degrees Cent. (930 degrees Fahr.) within the newly formed alpha needles may be seen from curve A in Fig. 73. We have found earlier that a hardness of C67 may be developed in the alloy by reheating at 600 degrees Cent. (1110 degrees Fahr.) for 1 hour following a water quench from 1300 degrees Cent. (2370 degrees Fahr.). In such a case, precipitation occurs in a matrix consisting entirely of the finegrained alpha solid solution formed from gamma during the water quench. A lightly etched microsection of the alloy fully hardened in this manner reveals, in Fig. 74, the original Widmannstätten pattern. If now but a part of the gamma had transformed before the 600 degrees Cent. (1110 degrees Fahr.) treatment we might expect rapid hardening only within those portions of the specimen occupied by the

Fig. 70—Alloy No. 1. Quenched from 1300 to 500 Degrees Cent., Held for 5 Hours and Water Quenched. × 200.

Fig. 71—Microcharacter Scratch on Section Shown in Fig. 70.

Fig. 72—Alloy No. 1. Quenched from 1300 to 500 Degrees Cent., Held for 50 Hours and Water Quenched. × 200.

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alpha phase, with a resultant hardness after one hour at 600 degrees Cent. (1110 degrees Fahr.) somewhat below the C67 value and bearing a close relation to the completeness of the gamma transformation. Curve B of Fig. 73 confirms this prediction at least in a qualitative measure. These hardness values were obtained from specimens which, after quenching to 500 degrees Cent. (930 degrees Fahr.),

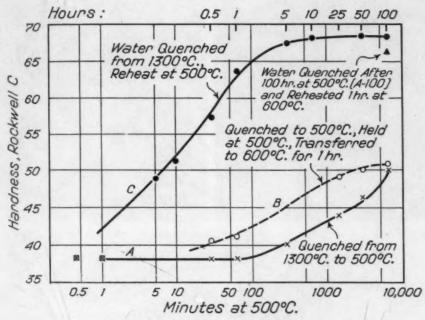


Fig. 73—Hardening at 500 Degrees Cent.

were held at this temperature for the periods indicated, then transferred directly to 600 degrees Cent. (1110 degrees Fahr.) and after an hour's heating quenched in water. Obviously, the formation of alpha must have started during the first few minutes at 500 degrees Cent. (930 degrees Fahr.), otherwise the subsequent treatment at 600 degrees Cent. (1110 degrees Fahr.) would neither have increased the hardness of the alloy nor yielded such a structure as we now see in Fig. 75. The result of the scratch test as pictured in Fig. 76 gives some idea of the hardening which has taken place within the needles during the treatment at 600 degrees Cent. (1110 degrees Fahr.). To indicate the normal hardening rate in a water quenched specimen when reheated at 500 degrees Cent. (930 degrees Fahr.) the curve C is included in Fig. 73.

Let us now consider the decomposition of gamma in a specimen at 450 degrees Cent. (840 degrees Fahr.) following a quench to that temperature from 1300 degrees Cent. (2370 degrees Fahr.). Ex-

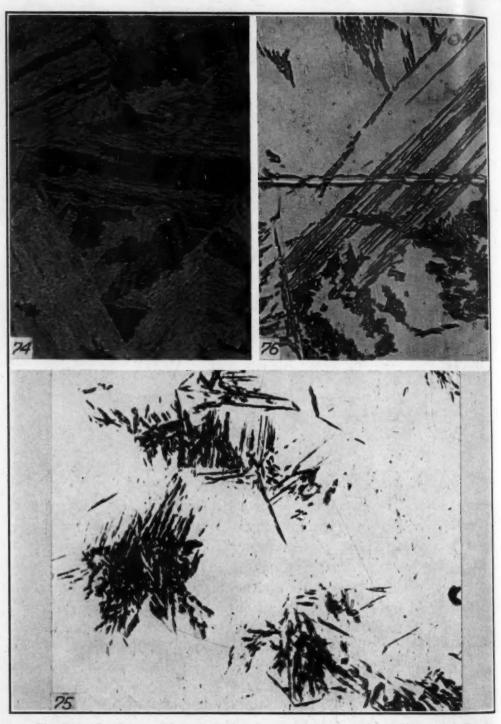


Fig. 74—Alloy No. 1. Water Quenched from 1400 Degrees Cent. Reheated 5 Hours at 600 Degrees Cent. to Maximum Hardness C 67. Etched 2 Seconds in 5 Per Cent Nital.

Fig. 75—Alloy No. 1. Quenched from 1300 to 500 Degrees Cent., Held for 5 Hours, Transferred Directly to 600 Degrees Cent., Held for 1 Hour and Water Quenched. Etched 2 Seconds in 5 Per Cent Nital.

Fig. 76—Microcharacter Scratch on Section Shown in Fig. 75.

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pansion begins almost immediately, according to the dilatometric data, and is substantially complete within 30 minutes. As may be seen from Fig. 77 the hardness also begins to increase without delay and continues to rise at a fairly uniform rate, reaching a value of C58 after 100 hours as indicated by curve A. The squares superposed on this curve are the values representing the "normal" age hardening

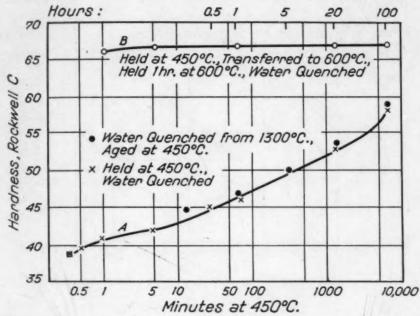


Fig. 77-Hardening at 450 Degrees Cent.

of this alloy at 450 degrees Cent. (840 degrees Fahr.) following a water quench from 1300 degrees Cent. (2370 degrees Fahr.). From the coincidence of these hardness values we may conclude that in both cases precipitation of the tungsten-rich phase is taking place within a fine-grained alpha matrix. We should, then, anticipate with confidence the rapid development of full hardness if a specimen after 1 hour at 450 degrees Cent. (840 degrees Fahr.) were to be transferred directly to 600 degrees Cent. (1110 degrees Fahr.) and held at this temperature for 1 hour, and curve B of Fig. 77 again fulfills our expectations.

While no attempt was made to determine the transformation-time relationships at temperatures below 400 degrees Cent. (750 degrees Fahr.) in this alloy, it is reasonably certain that in water quenching to room temperature the gamma is almost completely converted to alpha within a few seconds. Hardening data confirm this conclusion, for the maximum hardness is approached upon direct transfer to 600

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degrees Cent. (1110 degrees Fahr.) following a 5-minute period at 200 degrees Cent. No measurable hardening occurs at 200 degrees Cent. within a period of 100 hours and hence we must conclude that at this temperature the diffusion rate of tungsten is so low that precipitation and the attendant hardening occur very slowly, if at all.

Upon further consideration of the S curve in the light of the phenomena which have just been described, it seems probable that

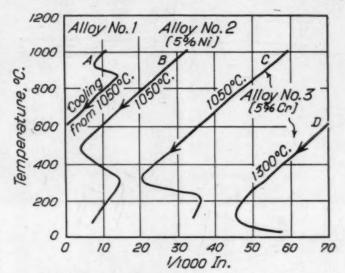


Fig. 78-Dilatometer Curves of Alloys No. 1, 2 and 3.

precipitation of the tungsten-rich phase precedes the allotropic transformation during the isothermal decomposition of the gamma solid solution at temperatures between 900 and 600 degrees Cent. (1650 and 1110 degrees Fahr.). In this temperature range, the precipitation of epsilon, by lowering the tungsten content of the gamma solid solution, accelerates the allotropic transformation. Below 500 degrees Cent. (930 degrees Fahr.) the rate of precipitation within the coarse-grained gamma has become so low that the allotropic transformation now occurs first, supplying a fine-grained alpha matrix within which precipitation can again proceed so long as the temperature is sufficiently high to permit diffusion of the tungsten. At about 300 degrees Cent. (570 degrees Fahr.) the rate of precipitation has become infinitely low while the formation of alpha on the other hand has probably approached completion within a few seconds. At temperatures between 500 and 600 degrees Cent. (930 and 1110 degrees Fahr.) the two reactions approach a deadlock and thus, within this temperature range, tend to preserve the gamma solid solution in a metastable state.

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At this point, it seems appropriate to recall a paper by DeLong and Palmer (13), published in 1928 in which was described the hardening of high speed by isothermal decomposition at about 600 degrees Cent. (1110 degrees Fahr.). In a discussion of this paper, Jeffries suggested as an explanation the possible precipitation of carbides and concurrent transformation of the austenite. Subsequent

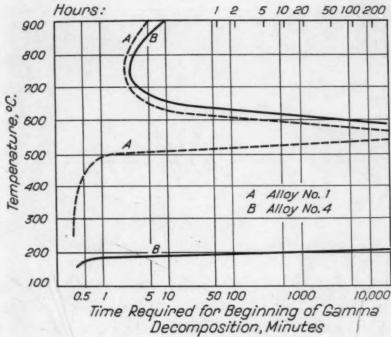


Fig. 79-"S" Curves of Alloys No. 1 and 4.

research has fully confirmed this opinion and we may now recognize in that phenomenon the behavior of the alloys in the present discussion.

Effects of Chromium, Nickel and Vanadium

Additions of nickel or chromium modify the behavior of the alloy No. 1 and in Fig. 78 we observe some dilatometric cooling curves which are comparable in cooling rate and size of specimen with those shown previously for the ternary alloy. The transformation is shifted to lower temperatures and as curve D demonstrates is strongly influenced by the temperature of the solution treatment.

To illustrate the similar effect of vanadium, we may use the "S" curve for alloy No. 4 shown in Fig. 79. This material differs from No. 1 mainly in the 2 per cent vanadium content. Here is seen an enormous increase in time elapsing before the onset of the

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temegrees this in a gamma transformation at temperatures between 600 and 200 degrees Cent. (1110 and 390 degrees Fahr.). After quenching to 200 degrees Cent. (390 degrees Fahr.) and holding for 100 hours, no hardening results from the transfer to 600 degrees Cent. (1110 degrees Fahr.) and hence we may conclude that no alpha has been formed within this interval. But still further, by lowering the tem-

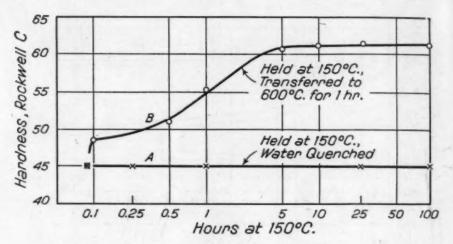


Fig. 80-Hardening Curves of Alloy No. 4 at 150 Degrees Cent.

perature of the quenching bath to 150 degrees Cent. (300 degrees Fahr.), the transformation begins without delay and its progress is reflected in the course of curve B in Fig. 80. The complete transformation of gamma in this alloy should insure an ultimate hardness of C 70 since such a hardness may be attained by aging at 600 degrees Cent. (1110 degrees Fahr.) following a water quench. Hence we may conclude that in this alloy the transformation approaches completion very slowly at 150 degrees Cent. (300 degrees Fahr.). Indeed the X-ray photograms from this material as water quenched from 1300 degrees Cent. (2370 degrees Fahr.) disclose the presence of retained gamma in appreciable amounts. After a solution treatment at 1400 degrees Cent. (2550 degrees Fahr.) both the nickel and chromium-bearing alloys (Nos. 2 and 3) either as air-cooled or water-quenched remain largely untransformed. The hardness is about C6 and increases only locally upon reheating at 600 degrees Cent. (1110 degrees Fahr.). After such a treatment the needles of alpha, within which precipitation has occurred, darken rapidly upon etching in nital while the structure of the gamma matrix is revealed as in Fig. 81 only by the application of some stronger etching reagent such as aqua regia.

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HARDNESS AT ELEVATED TEMPERATURES

We have seen the pronounced effect produced by cobalt upon the hardness of iron-tungsten alloys as determined at ordinary temperatures. It may, therefore, be fitting to mention here the results of a few hardness measurements made at 700 and 800 degrees Cent. (1290 and 1470 degrees Fahr.) upon a series of 20 per cent tungsten



Fig. 81—Alloy No. 3. 30 Per Cent Cobalt, 20 Per Cent Tungsten. 5 Per Cent Chromium, 45 Per Cent Iron. Water Quenched from 1450 Degrees Cent., Aged 1 Hour at 600 Degrees Cent. Etched First in 5 Per Cent Nital Then in Mixture of HNO₃, HCl, $\rm H_2O_2$ and Glycerol. $\rm \times$ 200.

alloys bearing cobalt in quantities ranging from 10 to 35 per cent. These tests were carried out at the Battelle Memorial Institute through the courtesy of Dr. Oscar E. Harder and his staff, following the procedure elsewhere described by Harder and Grove.

The alloys, previously treated to maximum hardness, were held at the temperatures of test for twenty minutes before the application of load. Perhaps the most striking feature of these data, plotted in Fig. 82, is the uniform rise in hardness at 700 degrees Cent. (1290 degrees Fahr.) which follows the increase in cobalt content up to 35 per cent. At 800 degrees Cent. (1470 degrees Fahr.) the relationship is apparently less direct, although the trend is undoubtedly the same. Harder and Grove (14) report a Brinell number of 300 in a

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high speed steel containing 6.7 per cent cobalt as tested at 700 degrees Cent. (1290 degrees Fahr.). While another high speed steel of 18-4-2 type showed a Brinell number of 260. They concluded that for this variety of steel, little, if any, increase in hardness at 700 degrees Cent. (1290 degrees Fahr.) could be expected from cobalt additions in excess of 5 per cent. In the case before us, the Brinell

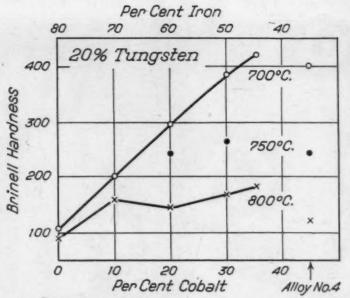


Fig. 82—High Temperature Hardness vs. Cobalt Content of Iron-cobalt-tungsten Alloys.

hardness of the 35 per cent cobalt alloy measures 415 which is at least 100 points in excess of any of the high speed steels tested at 700 degrees Cent. (1290 degrees Fahr.) by Harder and Grove. These observations suggest that in an iron base alloy cobalt most effectively increases the red hardness in the absence of carbon.

On the right-hand portion of the chart appear the Brinell hardness numbers shown by alloy No. 4 at the three temperatures of test. This material might be expected to compare favorably with the 30 per cent cobalt alloy which it closely resembles in composition. At 700 and 750 degrees Cent. (1290 and 1380 degrees Fahr.) such appears to be the case, but at 800 degrees Cent. (1470 degrees Fahr.) alloy No. 4 is distinctly the softer of the two. May this difference not result from the presence of the small amount of carbon in alloy No. 4?

At the extreme left in the diagram we encounter but slight softening in the binary iron-tungsten alloy as the testing temperature is raised from 700 to 800 degrees Cent. (1290 to 1470 degrees Fahr.).

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As will be recalled, this alloy represents a very coarse-grained alpha solid solution which had been previously hardened by a precipitation treatment at 700 degrees Cent. (1290 degrees Fahr.) and which overages but slowly at 800 degrees Cent. (1470 degrees Fahr.). Such behavior in this material under the present circumstances is therefore to be expected.

Conclusion

"To express it differently, the urge to age on the part of an excessively supersaturated solid solution of carbon in alpha iron is so great that aging starts at once, whereas in solid solution of nonferrous metals no such urge exists—hence time and even increased temperatures are required to cause precipitation."

Now, as we have seen today, some of these statements apply equally well to the carbon-free ternary alloys of iron, cobalt and tungsten. The gamma solid solution undergoes an allotropic transformation at a relatively low temperature during the quench. The solid solubility of tungsten in the newly formed alpha phase falls to about one quarter of its value at 1400 degrees Cent. (2550 degrees Fahr.). The microstructures resulting from gamma decomposition in the two alloys are remarkably similar. Precipitation of a carbide on the one hand and of a tungsten-rich compound on the other keeps pace with gamma-alpha transformation over limited ranges of decomposition temperatures. In the case of the steel this range extends down to 150 or 200 degrees Cent. In the other alloy precipitation begins to lag at 500 degrees Cent. (930 degrees Fahr.) and becomes infinitely slow at 300 degrees Cent. (570 degrees Fahr.). The behavior of both alloys when first quenched from the gamma

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slight erature Fahr.). range to room temperature and then reheated is represented by time-hardness curves almost identical in shape. But to react thus the carbon-free alloy must be aged at 525 to 600 degrees Cent. (975 to 1110 degrees Fahr.), whereas the steel requires a temperature of but 75 to 100 degrees Cent. (16). This difference may reasonably be considered to result directly from the slower diffusion rate of tung-

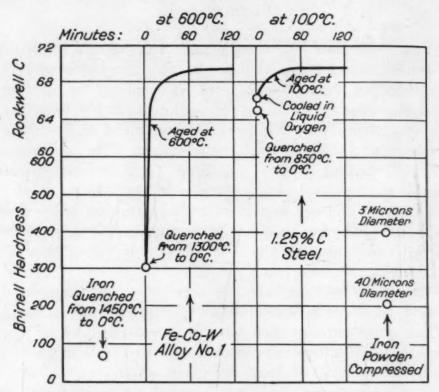


Fig. 83-Chart Comparing Several Methods of Hardening Iron.

sten in the alpha solid solution. As freshly quenched, both alloys exhibit the slow etching reaction typical of "white martensite," while the rapid darkening of the alpha needles by the etchant after aging indicates the presence of a highly dispersed precipitate.

One obstacle might yet seem to stand in the way of completing our analogy. I refer here to the considerable difference between the hardness of the carbon steel and that of the carbon-free ternary alloy as quenched to 0 degree Cent. from the gamma range. Let us discuss this apparent discrepancy with the aid of the chart in Fig. 83. As drastically quenched from 1450 degrees Cent. (2640 degrees Fahr.) a relatively pure iron shows a Brinell hardness of 70. In the ternary solid solution of cobalt and tungsten in iron after quenching from the same temperature we find a greatly increased hardness

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of 300 Brinell. Why then should we be overly surprised that even higher hardness is developed in the carbon steels by quenching from lower temperatures of the gamma range? Or that an additional hardness increase attends the conversion of retained austenite when the steel is subsequently further cooled to the temperature of liquid air? In this case both the smaller grain size of the gamma solid solution and the lower temperatures of its transformation might be expected to decrease the size of unit in the freshly formed alpha structure. Moreover, the mode of gamma transformation in the steel may differ from that existing in the ternary alloy and possibly further contribute to a greater hardness. Nor must we disregard the reasonable chance that alpha iron highly supersaturated with carbon may be much harder than the alpha solid solution of equal grain size in the ternary alloy.

An impressive demonstration of the close relation between hardness and particle size is furnished by some recent experiments of Dr. P. W. Bridgman and the speaker. Iron powders, hydrogen reduced from precipitated oxide were subjected to a pressure of 700,000 pounds per square inch. The particle diameter of the coarser powder averaged 40 microns while that of the finer averaged 3 microns. The compressed pellets were tested for hardness by both Rockwell and microcharacter methods. Expressed in Brinell numbers the hardness of the coarser metal measured about 200 while that of the finer approximated 400. When, therefore, the hardness of a metal may be increased from 70 to 400 Brinell by purely mechanical means is it out of all reason to expect a still greater hardening to result from an atomic rearrangement such as accompanies the formation of alpha from gamma at temperatures below 100 degrees Cent.?

In the upper portion of the diagram in Fig. 83 are shown the characteristic hardening curves of the two alloys which result from reheating after the quench. In the ternary alloy, this hardening accompanies precipitation of the tungsten-rich phase within the fragmented alpha matrix at 600 degrees Cent. (1110 degrees Fahr.). The carbon steel at 100 degrees Cent. arrives just as rapidly at the same ultimate hardness by a process which to all appearances is identical.

With due regard for our admittedly imperfect understanding of that property known as "hardness," this comparison seems to lead to the conclusion that precipitation of carbide is not essential

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to the hardness of a freshly quenched carbon steel. This statement may in turn be conditioned by the definition of the term "precipitation." With further refinements in terminology and experimental technique we shall doubtless approach more solid ground upon which to base future reasoning. In closing, may I express the hope that today's discussion shall have contributed in some little measure to our understanding of these hardening phenomena and encourage further effort in this field of investigation.

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FURTHER STUDY OF A HIGH CARBON-HIGH CHROMIUM TOOL STEEL

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431. tung der rungen," By W. H. WILLS

Abstract

This paper describes some physical tests run on an air hardening high carbon-high chromium tool steel. An investigation is made of: (1) the effect of various heat treatments on the impact value; (2) the effect of size of sections on the impact value following a given heat treatment.

The results show that prolonged heating at or above the normal hardening temperature reduces the impact value of the steel. Impact tests taken from larger sections show considerably lower impact value.

These results should be helpful in connection with the design and heat treatment of tools made from this type of steel.

INTRODUCTION

THE production of high carbon-high chromium tool steels has continued to grow so that they now represent important grades with the various tool steel mills. There are numerous analyses on the market, but they can conveniently be divided into two classes—the oil and the air hardening types. The oil hardening type carries a higher carbon content—usually from 2.00 to as high as 2.50 per cent—while air hardening runs about fifty points lower. The chromium contents of both types are about the same. The air hardening steels have approximately 0.75 to 1.00 per cent molybdenum, which imparts the air hardening property.

Although these steels do not develop as high a mineral hardness as straight carbon water hardening steels, they resist abrasion much better. They are also characterized by being deep hardening, low in impact value, good nondeforming quality, and are more difficult to machine than carbon or low alloy tool steels.

The history, metallography, and general physical properties of these steels are well covered by Gill¹ in a paper presented several

¹J. P. Gill, "High Carbon High Chromium Steels," Transactions, American Society for Metals, Vol. XV, No. 3, p. 387.

A paper presented before the Eighteenth Annual Convention of the Society held in Cleveland, October 19 to 23, 1936. The author, W. H. Wills, is associated with the metallurgical department of the Ludlum Steel Co., Dunkirk, N. Y. Manuscript received June 24, 1936.

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years ago. Also the author² in a previous paper discussed three analyses from the standpoint of manufacture, physical properties, and applications. These included —

	Steel	No. 1	Steel No. 2	Steel No. 3
Carbon	2.4	10 Per Cent	2.10 Per	r Cent 1.55 Per Cent
Manganese	0	35	0.30	0.30
Silicon	0	30	0.25	0.30
Chromium	11.5	50	12.00	12.00
Vanadium			1.00	0.25
Molybdenum	4			0.80

A study of the physical properties of these steels showed that so far as hardness is concerned, there is slight difference when hardened at the proper temperature with an oil quench. There is comparatively little falling off in hardness with draws up to 1000 degrees Fahr. (535 degrees Cent.). There is not much difference in compressive strength. Holding a comparatively long time at temperature has little effect on hardness structure, or dimensional changes. Steel No. 3 showed considerably higher impact values for all draws, and was the best as to nondeforming quality especially when hardened in air. With these advantages, along with ability to harden in oil or air and better machinability, the conclusion was that Steel No. 3 is the most universally adaptable of these three types.

In view of the widespread applications of the air hardening high carbon-chromium steels, and also certain points that were brought out in the discussion of this previous paper, it was thought desirable to go further with physical tests on this type.

The hardness and impact results given previously were run on test specimens hardened at one temperature within the standard hardening range of 1800 to 1850 degrees Fahr. (980-1010 degrees Cent.), holding for a comparatively short time at temperature, and quenching in oil. The series of draws ranged from 0 to 1000 degrees Fahr. (535 degrees Cent.). All test pieces were machined from ½ inch square hot-rolled bar stock to the usual size, 0.394 inch square (unnotched), and run on an Izod machine.

Later a few tests were run at 1800 degrees Fahr. (980 degrees Cent.) for a short and long time at temperature, and results showed a decrease in impact value when held a long time at temperature.

It was decided to investigate the effect on the hardness and impact value of this steel (1) following various heat treatments—test pieces all machined from one size, (2) following a single heat treatment—test pieces machined from sections of various sizes.

²W. H. Wills, "Practical Observations of Some High Carbon High Chromium Tool Steels," Transactions, American Society for Metals, Vol. XXIII, No. 2, p. 469.

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o. 3 Per Cent In this series it was attempted to cover a wider hardening range than is ordinarily encountered in the practical hardening of this grade. Thus three temperatures were selected—1700, 1800 and 1900 degrees Fahr. (925, 980, 1040 degrees Cent.), representing 100 degrees under the normal hardening temperature and 100 degrees over. The time at temperature was varied to include what would be used with tools of ordinary size. The drawing series was extended to 1200 degrees Fahr. (650 degrees Cent.), where in practice draws are usually not over 1000 degrees Fahr. (540 degrees Cent.).

(1) Effect of Heat Treatment

All test pieces were machined from ½ inch square hot-rolled annealed bar stock of analysis —

Carbon	1.56 Per Cent
Chromium	11.48
Vanadium	0.24
Molybdenum	0.76

They were planed to the standard size 0.394 inch square (unnotched) — four test pieces for each heat treatment.

There were six sets of tests, designated A to F inclusive, hardened as follows:

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A-1700 degrees Fahr. - ½ hour at temperature, open fire B-1800 degrees Fahr. - ½ hour at temperature, open fire C-1900 degrees Fahr. - ½ hour at temperature, open fire D-1700 degrees Fahr. - 3 hours at temperature, packed in spent charcoal E-1800 degrees Fahr. - 3 hours at temperature, packed in spent charcoal F-1900 degrees Fahr. - 3 hours at temperature, packed in spent charcoal
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All test specimens were cooled in still air. One lot of four from each set was not drawn. The others were given 2-hour draws in an electric furnace at respectively 300, 400, 500, 800, 1000, and 1200 degrees Fahr. (150, 205, 260, 425, 535, 650 degrees Cent.). The impact value was determined on an Izod machine and they were run for Rockwell C hardness. Results obtained are shown in Table I.

(2) Effect of Size of Section

The sizes selected included what would be considered ordinary or medium size for die work. The material was for the most part taken from stock on hand, and, while several melting heats were represented, there was comparatively little variation in the analysis. The sizes from which the impact tests were machined and the analyses represented were as shown under Table I, page 1018.

	Impact	Values		
Test No.	Heat Treatment Degrees Fahr.	Hardness Rockwell C	Ft. Lbs. Absorbed	Average Ft. Lbs.
A-1	1700 (1/2 hr.)-Air-No draw	57-59	47	- 41 4103,
A-2	do	do	39	
A-3	do	do	38	
A-4	do	do	39	40.8
A-5	1700 (½ hr.)—Air—300	58	37	
A-6 A-7	do	do	35	
A-8	do do	do - do	24 52	37.0
	Table of the Control of the Little of the Li			37.0
A-9 A-10	1700 (½ hr.)—Air—400 do	57-58 do	43	
A-11	do	do	22	
A-12	do	do	34	36.8
A-13	1700 (½ hr.)—Air—500	57-58	42	
A-14	do	do	39	
A-15	do	do	45	
A-16	do	do	30	39.0
A-17	1700 (1/2 hr.)—Air—800	54-56	23	
A-18	do	do	28	
A-19	do	do	36	10.
A-20	do	do	33	30.0
A-21	1700 (½ hr.)—Air—1000	50.5-51.5	52	1.00
A-22	do	do	50	
A-23	do	do	43	46.9
A-24	do	do	40	46.3
A-25 A-26	1700 (½ hr.)—Air—1200	36–38	76	
A-27	do do	do do	72 75	
A-28	do	do	56	69.8
B-1	1800 (1/2 hr.)-Air-No draw	63-64	43	
B-2	do	do	-42	
B-3	do	do	44	420
B-4	do	do	46	43.8
B-5	1800 (½ hr.)—Air—300	61-62.5	57	
B-6 B-7	do	do do	55	
B-8	do	do	53	56.3
B-9	1800 (½ hr.)—Air—400	59.5-60	55	
B-10	do	do	66	
B-11	do	do	52	
B-12	do	do	53	56.5
B-13	1800 (½ hr.)—Air—500	57-59	61	
B-14 B-15	do do	ob cb	70 40	
B-16	do	do	51	55.5
B-17	1800 (½ hr.)—Air—800	56-57.5	62	
B-18	do	do	65	
B-19	do	do	57	(20
B-20	do	do	67	62.8
B-21	1800 (½ hr.)—Air—1000	53-54.5	54	
B-22 B-23	do do	do do	51 70	
B-24	do	do	67	60.5
B-25	1800 (½ hr.)—Air—1200	38-39	110	
B-26	do	do	108	
B-27	do	do	98	105 2
B-28	do	do	105	105.3
C-1	1900 (1/2 hr.)—Air—No draw	63.5-64	24	
C-2 C-3	do do	do do	24	
C-4	do	do	23	24.5
C-5	1900 (½ hr.)—Air—300	62.5-63	43	
C-6	do	do	-26	
C-7	do	do	48	
C-8	do	do	38	38.8

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Test No. C-9 C-10 C-11 C-12 C-13 C-14 C-15 C-16 C-17 C-18 C-19 C-20 C-21 C-22 C-23 C-24 C-25 C-26 C-27 C-28 D-1 D-3 D-4 D-5 D-6 D-7 D-8 D-9 D-10 D-11 D-12 D-13 D-14 D-15 D-16 D-17 D-19 D-20 D-21 D-22 D-23 D-24 D-25 D-26 D-27 E-3 E-4 E-5 E-6 E-10 E-11 E-12 E-13 E-15 E-16 Average Ft. Lbs.

40.8

37.0

36.8

39.0

30.0

46.3

69.8

43.8

56.3

56.5

55.5

62.8

60.5

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Table I (Continued)

	Table I (C	ontinued)		
Test	Heat Treatment	Hardness	Ft. Lbs.	Average
No.	Degrees Fahr.	Rockwell C	Absorbed	Ft. Lbs.
C-9	1900 (½ hr.)—Air—400	61-61.5	67	
C-10	do	do	69	
C-11	do	do	66	
C-12	do	do	45	61.8
C-13	1900 (½ hr.)—Air—500	58–59	54	
C-14	do	do	55	65.5
C-15	do	do	70	
C-16	do	do	83	
C-17	1900 (½ hr.)—Air—800	57-58.5	71	
C-18	do	do	74	
C-19	do	do	67	
C-20	do	do	73	71.3
C-21	1900 (½ hr.)—Air—1000	58-58.5	42	43.3
C-22	do	do	40	
C-23	do	do	48	
C-24	do	do	43	
C-25 C-26 C-27 C-28	1900 (½ hr.)—Air—1200 do do	41.5–42.5 do do do	91 95 93 88	91.8
D-1	1700 (3 hrs.)—Air—No draw	57–57.5	23	71.0
D-2	do	do	31	
D-3	do	do	29	
D-4	do	do	42	31.3
D-5	1700 (3 hrs.)—Air—300	56.5–58	21	
D-6	do	do	39	
D-7	do	do	27	27.8
D-8	do	do	24	
D-9 D-10 D-11 D-12	1700 (3 hrs.)—Air—400 do do	56-57 do do	37 31 44	25.0
D-12 D-13 D-14 D-15	1700 (3 hrs.)—Air—500 do	do 56–56.5 do do	31 28 19 27	35.8
D-16	do	do	41	28.8
D-17	1700 (3 hrs.)—Air—800	53-54.5	46	
D-18	do	do	30	
D-19 D-20 D-21 D-22	do do 1700 (3 hrs.)—Air—1000	do do 51-52	30 20 45	31.5
D-23 D-24 D-25	do do do 1700 (3 hrs.)— Air —1200	do do do 34–35.5	54 49 40 108	47.0
D-26	do	do	84	94.0
D-27	do	do	110	
D-28	do	do	74	
E-1 E-2 E-3 E-4	1800 (3 hrs.)—Air—No draw do do	63.0-64 do do	19 17 19	
E-5 E-6 E-7	do 1800 (3 hrs.)—Air—300 do do	do 62–62.5 do	17 23 20 22	18.0
E-8	do	do	20	21.2
E-9	1800 (3 hrs.)—Air—400	59.5–60.5	14	
E-10 E-11 E-12	do do do	do - do	30 17	20.1
E-13 E-14 E-15	1800 (3 hrs.)—Air—500 do	do 57.5–59 do	20 32 18	20.1
E-16	do do	do do	16 20	21.5

Test	Heat Treatment	Hardness	Ft. Lbs.	A
No.	Degrees Fahr.	Rockwell C	Absorbed	Average Ft. Lbs.
E-17	1800 (3 hrs.)—Air—800	57-58	29	2 6, 1,03,
E-18	do	do	35	
E-19	do	do	36	
E-20	do	do	30	32.5
E-21	1800 (3 hrs.)—Air—1000	53-54.5	37	
E-22	do	do	24	
E-23 E-24	do do	do do –	. 25	20.0
E-25				30.0
E-25 E-26	1800 (3 hrs.)—Air—1200 do	40-41 do	30 62	
E-27	do	do	62	
E-28	do	do	54	52.0
F-1	1900 (3 hrs.) - Air-No draw	61-62	16	,
F-2	do	do	15	
F-3	do	do	13	
F-4	do	do	14	14.5
F-5	1900 (3 hrs.)—Air—300	59-62	22	
F-6 F-7	do	do	15	
F-8	do	do do	21 15	18.3
F-9	1900 (3 hrs.)—Air—400	56-61.5	25	10.3
F-10	do	do	34	
F-11	do	do	20	
F-12	do	do	28	26.8
F-13	1900 (3 hrs.)—Air—500	57-59	22	
F-14	do	do	30	
F-15 F-16	do	do	20	04.3
	do	do	25	24.3
F-17 F-18	1900 (3 hrs.)—Air—800	57-59.5 do	26 23	
F-19	do	do	34	
F-20	do	do	34	29.3
F-21	1900 (3 hrs.)—Air—1000	61-63	23	
F-22	do	do	25	
F-23	do	do	24	
F-24	do	do	20	23.0
F-25	1900 (3 hrs.)—Air—1200	46-47	35	
F-26	do	do	42	
F-27 F-28	do	do	31 32	35.0
F-28	do	do	34	33.0

		Analysis-Per Cent			
	Size	C	Cr	V	Mo
Squares and Flats	\begin{cases} \frac{1\frac{1}{2}''}{4''} & \text{sq.} \\ 6'' & \text{x} & \frac{1}{2}'' \\ \end{cases} \text{6''} & \text{x} & \text{2'\frac{1}{2}''} \end{cases}	1.56 1.56 1.49	11.53 11.53 11.44	0.25 0.25 0.26	0.79 0.79 0.82
Rounds	7%" dia. 2 " dia. 35%" dia. 5 " dia.	1.53 1.49 1.53 1.53	12.15 11.50 11.67 11.67	0.25 0.22 0.22 0.22	0.73 0.82 0.75 0.75
Upset Forgings	\[\begin{pmatrix} 4'' & dia. x & \frac{1}{2}'' & thick \\ 6'' & dia. x & 1 & '' & thick \\ 8'' & dia. x & 2 & '' & thick \end{pmatrix} \]	1.54	11.48 (all from sam	0.24 ne heat)	0.76

From each of the above sections, four standard size 0.394 inch square unnotched impact test pieces were machined. In the larger sizes of flats and rounds they were cut representing, so far as possible, the midsection between the center and exterior. The direction of the grain of the metal in the test specimens was longitudinal, and thus

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Average Ft. Lbs.

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Mo

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0.82

0.73 0.82 0.75 0.75

0.76

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Table II Impact Values

	Impact	alues		
Test No.	Machined from Section	Hardness Rockwell C	Ft. Lbs. Absorbed	Average Ft. Lbs.
G-1	½" sq.	59.5-60	69	
G-2	do	do	64	
G-3	do	do	77	
G-4	do	do	43	63.3
H-1	4" x 3/4"	59-59.5	51	
H-2	do	do	37	
H-3	do	do	61	
H-4	do	do	56	51.3
I-1	6" x 21/2"	58-59.5	42	
I-2	do	do	31	
I-3	do	do	40	
I-4	do	do	44	39.3
T-1	7/8" rd.	58.5-59	62	
J-2	do	do	44	
J-3	do	do	66	
J-4	do	do	64	59.0
K-1	2" rd.	58-59.5	67	
K-2	do	do	58	
K-3	do	do	63	
K-4	do	do	53	60.3
L-1	35%" rd.	58-58.5	38	
L-2	do	do	32	
L-3	do	do	25	20.2
L-4	do ,	do	34	32.3
M-1	5" rd.	58.5-59	28	
M-2	do	do	24	
M-3	do	do	24	05.5
M-4	do	do	26	25.5
N-1	Forging-4" dia. x 1/2" thick	58-60	61	
N-2	do	do	42	
N-3	do	do	26	
N-4	do	do	31	40.0
0-1	Forging-6" dia. x 1" thick	59-59.5	10	
0.2	do	do	8	
0.3	do	do	8	
0-4	do	do	14	10.0
P-1	Forging—8" dia. x 2" thick	58-58.5	9	
P-2	do	do	9	
P-3	do	do	10	
P-4	do	do	10	9.5

similar to that of the bar stock. In the case of those from the forgings, the direction of the grain was transverse to that of the upset grain structure.

It was decided to subject these test specimens to a heat treatment involving a uniform time at the hardening temperature, rather than attempt to duplicate the exact time that would ordinarily be required for the individual sections. This represented roughly a mean between the time that would apply for the largest and smallest. This heat treatment was as follows:

The test pieces were heated in an electric furnace with a slightly reducing atmosphere, so that scaling was held to a minimum. After bringing up to 1800 degrees Fahr. (980 degrees Cent.), they were

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held one hour at temperature, removed, and cooled in still air, then drawn in an electric furnace at 400 degrees Fahr. (205 degrees Cent.) for two hours.

The impact value was determined on an Izod machine, and the Rockwell C hardness checked similar to the previous groups. These results are shown in Table II.

DISCUSSION OF RESULTS

It will be noted that in some cases there is considerable variation in the impact values obtained for a given heat treatment. The method used cannot be considered very accurate on this kind of material, although results are useful for purposes of comparison and

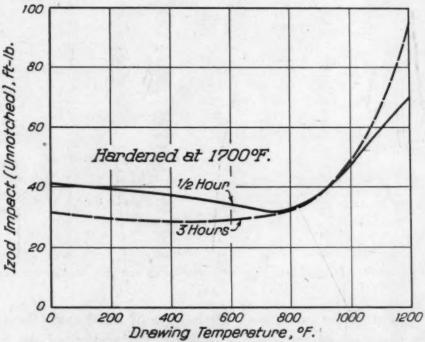


Fig. 1—Air Hardening High Carbon-High Chromium Tool Steel Impact Values Versus Drawing Temperature. Curve 1 Hardened 1700 Degrees Fahr., ½ Hour. Curve 2 Hardened 1700 Degrees Fahr., 3 Hours.

numerous previous tests had been run with this equipment. There might be a possible source of error in carburization or decarburization of the test specimens. The condition was checked up by microexamination and the maximum decarburization observed was approximately 0.005 inch. There was not any evidence of carburization.

The initial hardness developed by this steel when hardened at 1700 degrees Fahr. (925 degrees Cent.), holding for ½ hour and r, then Cent.)

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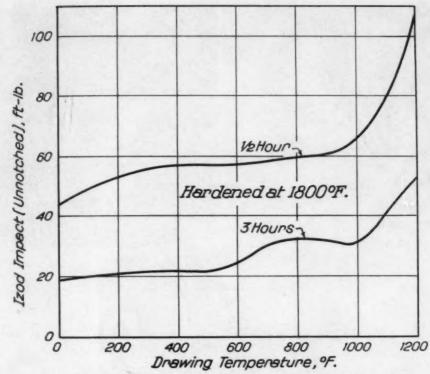


Fig. 2—Air Hardening High Carbon-High Chromium Tool Steel Impact Values Versus Drawing Temperature. Curve 1 Hardened 1800 Degrees Fahr., ½ Hour. Curve 2 Hardened 1800 Degrees Fahr., 3 Hours.

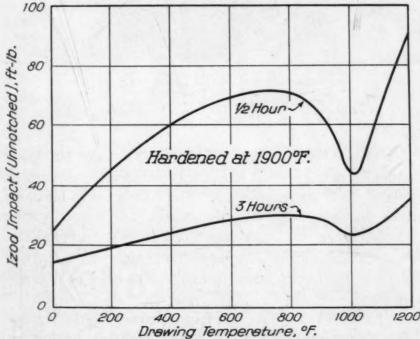


Fig. 3—Air Hardening High Carbon-High Chromium Tool Steel Impact Values Versus Drawing Temperature. Curve I Hardened 1900 Degrees Fahr., ½ Hour. Curve 2 Hardened 1900 Degrees Fahr., 3 Hours.

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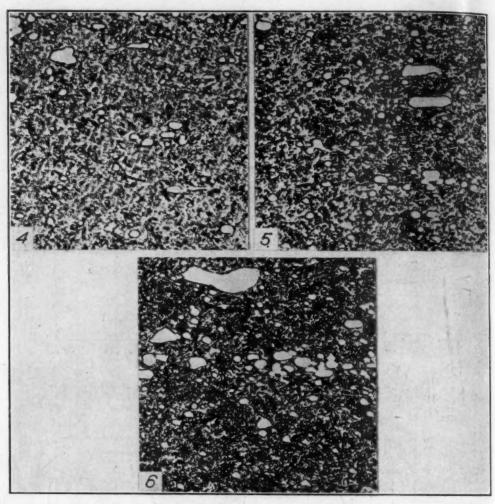


Fig. 4—Structure Typical of Test No. A-1 and D-1. Fig. 5—Structure Typical of Test No. A-3 and D-3. Fig. 6—Structure Typical of Test No. A-6 and D-6. Hardened at 1700 Degrees Fahr., All Magnification × 600.

3 hours at temperature, is approximately the same, but lower than when hardened at the normal hardening temperature — namely, 1800 degrees Fahr. (980 degrees Cent.). The falling off in hardness after the various draws is practically the same. The impact value developed is slightly lower than when hardened at 1800 degrees Fahr. (980 degrees Cent.) and is little affected by the length of time at this comparatively low hardening temperature (1700 degrees Fahr.) — see curves of Fig. 1.

When hardened at the normal temperature — 1800 degrees Fahr. (980 degrees Cent.) — with the short and long time held at temperature, the initial hardness is practically the same as is also the hardness after the various draws. The best average impact values were ob-

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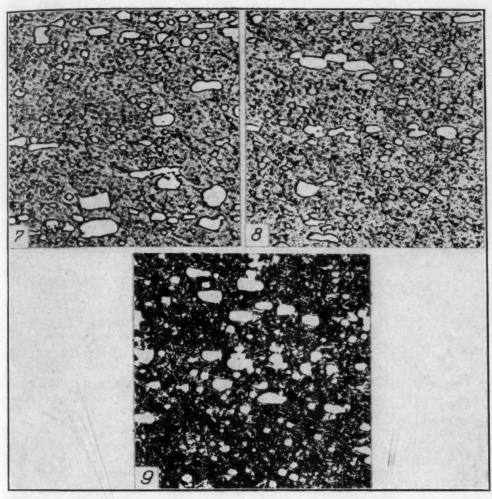


Fig. 7—Structure Typical of Test No. B-1 and E-1. Fig. 8—Structure Typical of Test No. B-3 and E-3. Fig. 9—Structure Typical of Test No. B-6 and E-6. Hardened at 1800 Degrees Fahr., All Magnification × 600.

tained when holding the short time at 1800 degrees Fahr. (980 degrees Cent.). The effect of holding a long time (3 hours) at 1800 degrees Fahr. (980 degrees Cent.) was to reduce the impact values considerably as shown by Fig. 2.

The effect of holding for a short time at 1900 degrees Fahr. (1040 degrees Cent.) was much the same as at 1800 degrees Fahr. (980 degrees Cent.). Holding 3 hours at 1900 degrees Fahr. (1040 degrees Cent.) resulted in slightly lower initial hardness, but distinct secondary hardness after the draw at 1000 degrees Fahr. (535 degrees Cent.). The reduction in impact value after the long time at 1900 degrees Fahr. (1040 degrees Cent.) was quite pronounced — see Fig. 3.

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In order to make comparison of the structures developed by these various treatments, microexamination was made of longitudinal sections from some of the impact test pieces. These included one from each set as quenched, drawn at 400 degrees Fahr., and drawn at 1000 degrees Fahr. (535 degrees Cent.). No appreciable difference was observed between the structures of those held the short and long periods at temperature, and they are listed together as follows:

Test No.	Heat Tre Degrees		Structure
A & D-1 (Fig. 4)	1700 (½ & 3 hrs.)	No draw	Mixed austenite and marten- site with numerous small un- dissolved carbides.
A & D-3 (Fig. 5)	do do	Draw 400	Mixed austenite with larger proportion of martensite. Numerous small undissolved carbides.
A & D-6 (Fig. 6)	do do	Draw 1000	Mixed martensite and troost- ite. Numerous small undis- solved carbides.
B & E-1 (Fig. 7)	1800 (½ & 3 hrs.)	No draw	Largely austenitic. Slight trace of grain boundaries.
B & E-3 (Fig. 8)	do do	Draw 400	Mixed austenite and marten- site. Slight trace of grain boundaries.
B & E-6 (Fig. 9)	do do	Draw 1000	Fine mixed martensite and troostite.
C & F-1 (Fig. 10)	1900 (½ & 3 hrs.)	No draw	Austenitic. Grain boundaries pronounced.
C & F-3 (Fig. 11)	do do	Draw 400	Austenitic with some martensite. Grain boundaries pronounced.
C & F-6 (Fig. 12)	do do	Draw 1000	Slightly coarse. Mixed martensite and troostite.

The test specimens from lots G to P inclusive which received a uniform heat treatment showed, of course, essentially the same structure—mixed austenite with some martensite and slight trace of grain boundaries—as specimen B-3. However, due to the different size sections, the size and distribution of the carbides varied. An examination of longitudinal sections of these showed the following:

Test Lot	Original Size	Remarks as to Carbide Distribution
G	1/2" sq.	Quite uniform. Slight tendency for arrangement in lines.
H	4" x 3/4"	Some tendency toward carbide segregation in lines.
I	6" x 2½"	Some tendency toward carbide network rather than arrangement in lines.

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Fig. 10—Structure Typical of Test No. C-1 and F-1. Fig. 11—Structure Typical of Test No. C-3 and F-3. Fig. 12—Structure Typical of Test No. C-6 and F-6. Hardened at 1900 Degrees Fahr., All Magnification × 600.

*	7/8" rd.	Quite uniform—similar to lot G.
	2" rd.	Some tendency toward carbide segregation in lines.
	35/8" rd.	Slight tendency toward carbide network.
	5" rd.	Carbide network more pronounced than lot L.
	4" x ½" forging	Rather uniform with little carbide segregation.
	6" x 1" forging	Some tendency toward carbide network.
	8" x 2" forging	Carbide network rather pronounced.
	*	2" rd. 35%" rd. 5" rd. 4" x ½" forging 6" x 1" forging 8" x 2"

It is rather difficult to explain the variations of impact value exhibited by this steel with these different heat treatments. The complex structures developed do not simplify the problem. There is ap-

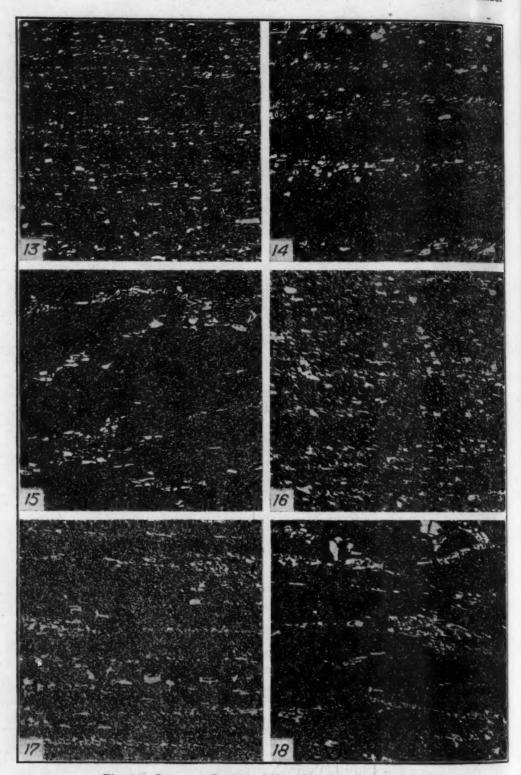


Fig. 13—Structure Typical of Test Lot G. ½ Inch Square.
Fig. 14—Structure Typical of Test Lot H. 4 x ¾ Inch.
Fig. 15—Structure Typical of Test Lot I. 6 x 2½ Inch.
Fig. 16—Structure Typical of Test Lot J. ¼ Inch Round.
Fig. 17—Structure Typical of Test Lot K. 2 Inch Round.
Fig. 18—Structure Typical of Test Lot L. 35% Inch Round. All × 125.

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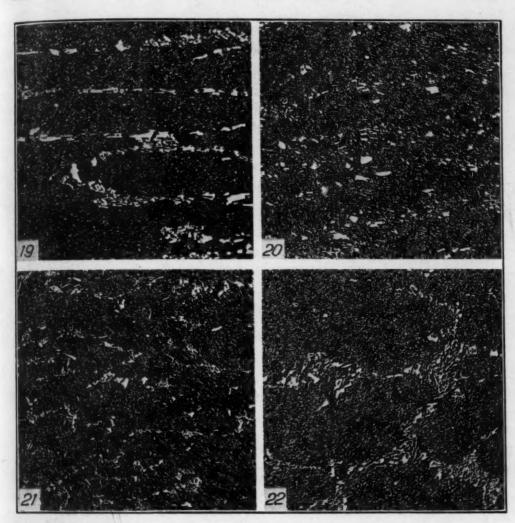


Fig. 19—Structure Typical of Test Lot M. 5 Inch Round.

Fig. 20—Structure Typical of Test Lot N. 4 Dia. x ½ Inch Thick Forging.

Fig. 21—Structure Typical of Test Lot O. 6 Dia. x 1 Inch Thick Forging.

Fig. 22—Structure Typical of Test Lot P. 8 Dia. x 2 Inch Thick Forging.

All Photomicrographs. × 125.

parently a certain proportion of austenite and martensite that produces a structure of high hardness and relatively high impact value as quenched. When such is the case, there is a gradual increase of impact value following increasing drawing temperatures.

If the proportion of austenite present in the structure as quenched is relatively low, the impact value is well maintained but is not much affected by drawing until rather high temperatures are reached—1000 degrees Fahr. and over. If on the other hand the structure as quenched is largely austenitic, the impact value is decreased. When such is the case, draws up to and including 800 degrees Fahr. improve the impact value, but there is a limited range

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around 1000 degrees Fahr. where there is a decrease. With draws beyond this the impact value is increased.

It is worthy of note that the austenitic structure developed by this type of steel when hardened at the upper temperatures (1800 and 1900 degrees Fahr.) is somewhat analogous to that which is characteristic of high speed steel. This is broken down into martensite, troostite, etc., with increasing drawing temperatures and the impact value is affected in much the same manner.

As would naturally be expected, the impact tests taken from the larger sections, both flats and rounds, showed materially less toughness than those from the small sizes. Within the range tested this decrease was roughly about 50 per cent. The tests from the upset forgings showed up lower than was expected, particularly those from the larger sizes.

Conclusions

The results of this series of tests should be of some use in connection with the design and heat treatment of tools made of this type of steel. While a relatively long heating period is required in hardening as compared to carbon or low alloy tool steels, it is possible to go too far and hold too long at the hardening temperature particularly when pack hardening, and the temperature of the charge is judged by experience. It has been definitely shown that this results in lower impact value.

Steels of this type have a rather wide hardening range and may be held at the hardening temperature a comparatively long time without impairing any of their properties. The ideal time factor would of course be the least period necessary to develop maximum hardness as quenched. This is a difficult thing to state definitely, due to the widely different rates of heating in various kinds of furnaces. The use of furnaces with definitely controlled atmospheres makes it possible to harden these steels without packing, which is a distinct advantage in the saving of time and in better knowledge of the proper time at temperature.

The high carbon-high chromium steels are subject to more or less carbide segregation, even to a greater extent than high speed steels. The ingot structure is characterized by the same network or cellular arrangement of the carbide segregate. This is broken up only by the mechanical working of the steel. There is, therefore, considerably

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or less steels. ellular by the erably more uniform distribution of the carbides in small sections such as $\frac{1}{2}$ inch square as compared to sizes 3 inches round or larger. Consequently it is not surprising that the impact value of the larger sections such as $6 \times 2\frac{1}{2}$ inches, $3\frac{5}{8}$ inches round and 5 inches round was considerably less than that of the smaller sections.

These steels have been used successfully on numerous and varied applications, some involving considerable shock. When the latter is the case, it is important that due consideration be given to the design in providing an ample factor of safety with reference to maximum stresses involved. At the same time, excessively large sections should be avoided so far as possible by building up in sections.

ACKNOWLEDGMENTS

The author wishes to express his thanks to F. B. Lounsberry, Vice-President of the Ludlum Steel Company, for permission to present the data given in this paper. Special credit should be given to L. R. Lounsberry of the metallurgical laboratory for his part in conducting the various physical tests and for the metallographic work involved.

RECOVERY OF COLD-WORKED NICKEL AT ELEVATED TEMPERATURES

By ERICH FETZ

Abstract

The preparation of highly pure metals led in many cases to new physical property values, as for instance hardness. The marked effect of impurities is also noticed in regard to the softening ("recovery") of cold-worked metals on annealing. Experiments with 99.866 and 99.99 per cent pure nickel yielded a considerably lower softening temperature range than 12 former investigations to be found in the international literature. With highly pure, cold-rolled nickel, the following phenomena occur in this order in different temperature ranges: sharpening of the Debye-lines, recovery of hardness, recrystallization. Increasing the reduction of thickness by 10 per cent lowers linearly the softening temperature of nickel by 23.3 degrees Cent.

A redetermination of the recovery of physical properties of plastically deformed, highly pure metals is not only of scientific interest, but also of considerable technological importance in view of the constantly advancing purities of commercially available metals.

ALTHOUGH cold-working as a means of increasing the strength of metallic materials has been applied for centuries, science to date has failed to furnish a satisfactory explanation for this process and for the restoration of the initial physical and chemical properties on annealing. Atomic physics has not yet bridged the gap between atomic structure or arrangement (diamond-graphite-carbon!) and hardness; the extension of the mathematical theory of elasticity to finite deformations is still in its infancy; it cannot even be decided at present whether the various methods of increasing the hardness by alloy additions, cold-working, electrolytic deposition, lattice transformation, precipitation-hardening, hot pressing of metal powders, etc., are in principle due to the same physical cause.

From a technological point of view, work-hardening is the most

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A paper presented before the Eighteenth Annual Convention of the Society held in Cleveland, October 19 to 23, 1936. The author, Erich Fetz, is research metallurgist, Wilbur B. Driver Co., Newark, N. J. Manuscript received May 12, 1936.

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important physical change produced by plastic deformation and experimental and theoretical research has concentrated therefore on the investigation and interpretation of this phenomenon. However, discussion of the hardness theories of Beilby, Polanyi, Heyn, Sachs, Tammann, Ludwig, Jeffries and Archer, Dean and Gregg, Czochralski, Hencky, Stepanow, Wood, Kornfeld and others will be omitted here, as X-ray analysis refuted several theories in principle and some investigators abandoned their earlier hypothesis in view of new experimental evidence.

After van Arkel¹ established by X-rays that cold-working produces but slight parameter changes, which, for instance, could hardly be the cause of the drastic changes of electrical conductivity, Geiss and van Liempt² advanced the concept that the atoms themselves, i.e., their outer electron shells, are affected by cold work. This may or may not be accompanied by lattice distortions. The systematic research of Tammann and co-workers⁸ confirmed that physical changes due to cold work, and their reversion on annealing depend not only on the lattice type but also on the position in the periodic system of the atoms building up the lattice. Although copper, aluminum, nickel and platinum possess the same cubic face-centered space lattice, remarkable differences occur during strain hardening and recovery therefrom. As the author recently proved,4 strain hardening and precipitation hardening effects, contrary to previous conceptions of Dahl,⁵ can be superimposed, a definite corroboration of the theory that cold work affects the atoms themselves.

At elevated temperatures, the structure produced by cold work is replaced by the familiar recrystallization visible under the microscope. Lattice distortions, which may manifest themselves in a broadening of the Debye lines, are also eliminated and the same X-ray picture is obtained as before cold-working. The hypothesis that the restoration of the physical properties of the annealed state is closely related to recrystallization cannot be upheld. An especially large temperature gap between softening (800 degrees Cent.) and recrystallization (1500 degrees Cent.) was observed on tungsten wire.

¹A. E. van Arkel, Physica, Vol. 5, 1925, p. 208.

²W. Geiss and J. A. M. van Liempt, Z. anorg. Chem., Vol. 133, 1924, p. 107; Vol. 143, 1925, p. 259; Vol. 195, 1931, p. 366; Z. Metallk., Vol. 18, 1926, p. 216; Z. Physik, Vol. 45, 1927, p. 631.

⁸G. Tammann, Z. Metallk., Vol. 26, 1934, p. 97; Vol. 28, 1936, p. 6.

⁴E. Fetz, Z. Physik, Vol. 97, 1935, p. 690.

⁵O. Dahl, Wiss. Veröff. Siemens Kons.. Vol. 8, 1929, p. 157.

⁶F. Koref, Z. Metallk., Vol. 17, 1925, p. 213.

Copper can be work hardened by forging well over 700 degrees Cent.¹

although it recrystallizes at about 150 degrees Cent. The recovery

from cold- and hot-working is a phenomenon far more complicated

than was originally assumed and unfortunately experimental data are

meager and contradictory. Because hardness recovery takes place

at elevated temperatures, its possible relation to the mobility of the

atoms in the space lattice appeared to be logical. But in spite of a

considerable lowering of the melting point by the addition of an

alloying element, the softening temperature range is often raised

materially. Contaminations may act likewise. Pure, cold-worked copper completely softens at 80 degrees Cent. after 41 days,8 but

only 0.022 per cent silver causes copper to be almost entirely unre-

lieved of its strain hardening after 365 days at 150 degrees Cent.⁹

In view of this marked effect of contamination upon the recovery of

"pure" copper—and to a greater extent upon that of nickel—and be-

cause recovery depends also on amount, method, temperature (tin)10

and speed of cold-working, time of annealing, pretreatment and or-

igin of testing material, it is not surprising that the findings of vari-

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Elimination by Annealing of Physical Property Changes
Due to Cold-Working of Nickel

The internal energy stored by cold-working nickel is released in two temperature ranges on heating. According to Satô,¹¹ the first heat evolution coincides with the Curie point, while the rest of the latent energy is released at 630 and 660 degrees Cent. depending on twisting. Nickel loses its ferromagnetism between 340 and 360 degrees Cent. The electrical resistance partially recovers at 350 degrees Cent.¹² The recovery curve of elasticity changes its direction at about 300 degrees Cent., that of the reverse bending number at 440 degrees Cent. and of the bending resilience at 480 degrees Cent.¹³ Thermoelectric force recovers in the same temperature range as electrical resistance.¹⁴ The recovery curves of magnetic induction fall

ous investigators show considerable deviation.

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⁷F. Sauerwald, Metallwirtsch., Vol. 7, 1931, p. 1353.

⁸A. v. Zeerleder and P. Bourgois, Journal, Institute of Metals, Vol. 62, 1929, p. 321.
⁹H. C. Kenny and G. L. Craig, American Institute of Mining and Metallurgical Engineers, Technical Publication No. 525.

¹⁰A. E. van Arkel and Ploos van Amstel, Z. Physik, Vol. 62, 1930, p. 43.

¹¹S. Satô, Science Reports, Tôhoku Imperial University, (I) Vol. 20, 1931, p. 140.

¹²G. Tammann and G. Moritz, Ann. Physik, (5) 16, 667, 1933.

¹⁸K. L. Dreyer and G. Tammann. Heraeus Vakuumschmelze 1923/33, p. 86.

¹⁴G. Tammann and G. Bandel, Ann. Physik, (5) 16, 120, 1933.

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off between 590 and 620 degrees Cent. 15 Thermal resistance begins to recover at about 200 degrees Cent. Unfortunately, Tammann and Böhme¹⁶ did not obtain with nickel a well shaped recovery curve with two distinct horizontal sections as they did with iron, copper and silver. The same holds for the recovery curve of the temperature coefficient.12 The recovery curve of the modulus of rigidity17 and Young's modulus of elasticity¹⁸ pass through a maximum at 350 degrees Cent. The former begins to drop below the initial value at 560 degrees Cent. and the latter at 420 degrees Cent. The bending endurance strength of cold-worked nickel falls off only after annealing above 600 degrees Cent.19 Hardness and tensile strength curves follow apparently the same course. According to Ludwig,20 both measure the same physical property, viz., a mean resistance to deformation, of those metals which show a marked reduction of area. yield point seems to recover at lower temperatures than tensile strength, whereas elongation and reduction of area recover simultaneously with hardness. Dreyer and Tammann¹³ state that "hardness recovers only after complete recrystallization," which they found at 600 degrees Cent. This appears to be very doubtful. According to the author's experiments, 21 addition of 15 or 20 per cent nickel shifts the softening point of copper to over 500 degrees Cent. If the findings of Tammann and Moritz,12 that 60 per cent cold-worked nickel recovers at 670 degrees Cent., were correct, the recovery temperature of nickel would be lowered by alloying copper with it.

RECOVERY OF HARDNESS AND TENSILE STRENGTH OF COLD-WORKED NICKEL

Literature statements on the softening of cold-worked nickel show unusually great disagreement (see Table I). An evaluation of the recovery curves of nickel reduced more than 50 per cent shows differences of 400 degrees Cent. The majority of the recovery curves show pronounced softening around 600 degrees Cent.* A surpris-

¹⁵G. Tammann and H. J. Rocha, Ann. Physik, (5) 16, 861, 1933.

¹⁶G. Tammann and W. Böhme, Ann. Physik, (5) 22, 500, 1935.

 ¹⁷T. Kawai, Science Reports, Tôhoku Imperial University, (I) Vol. 20, 1931, p. 681.
 ¹⁸T. Kawai, Science Reports, Tôhoku Imperial University, (I) Vol. 19, 1930, p. 209.

¹⁹Nickelhandbuch, Frankfurt-Main, Sect. "Nickel," p. 18.

²⁰P. Ludwig, Z. Metallk., Vol. 14, 1922, p. 101. ²¹E. Fetz, Metallwirtsch., Vol. 15, 1936, p. 167.

^{*}In the following, the turning point Tr of the recovery curve is used as a characteristic value, i.e., that point on the curve where a tangent changes direction.

Temp.	Anneal. Time	Reduc- tion in %	Method of Cold Working	Physical Property Tested	Analysis of Nickel	Experimenter	Year	Remarks
009	w	۵.	Hammering	Hardness	*.%666	Matweef	11611	*Information by Dr. G. M. Möhling
550	30	72.3	Rolling	Hardness*	99,58% Mond Ni	Rose	1912	*Rebound hardness
069	3	*09	Drawing	Tensile Strength	a.	Guillet	1913	*Estimated
620	20*	73	Rolling	Tensile Strength	98.48% Ni + Co, 0.67% Fe, 0.27% Mn, 0.162% Si, 0.22% Cu, 0.07% C, 0.033% S	Price and Davidson	1920	*25 minutes pre-
510	09	50	Upsetting	Hardness	% Mn, 0.0	Schottky and Jungbluth	1923	
590	۸.	*04	Drawing	Tensile Strength	99.01% Ni, 0.39% Co, 0.17% Cu, 0.1% Co, 0.11% C, 0.011% P, 0.128% Fe, 0.16% Mn, 0.026% Si, S + Al = 0	Mochel	1928	*Estimated
740	See text	20	Rolling	Hardness	a.	Crawford and Worthington	1930	-
650	30	31	Drawing	Tensile Strength	99.5% Mond Ni	Kawai	1931	
009	S	75	Drawing		, 0.45% C			
410	S	20	3	Tensile Strength		Ransley and Smithells	1932	
550 550 620	เกเกเก	70 20 20	5 5 5 5 5 5		ditto + 0.34% Mn ditto + 0.07% Mg		**	
089	09	53	Drawing	Tensile Strength	% Fe	Ancelle	1933	3
029	30	06	Rolling	Hardness*	99.1% Ni (Ni used by Tammann and Bandel ¹⁴)	Tammann and Moritz	1933	*Indentations converted into pene- tration hardness in kg/mm²
580	09	7.5	Upsetting	Hardness	99.8% Ni, <0.05% C*	Wilson and Thomassen	1934	*Information by Dr. Wilson
800	30	۵.	Drawing	Hardness	0.44% Co, 0.16% C, 0.12% Mn, 0.18% Fe, 0.01% Si, 0.03% Cu, bal. Ni	Ellis	1934	
440 454 463	30	30 80 F	Rolling	Hardness	0.11% Co, 0.013% Fe, trace Si + S. Electrolytic Ni, as deposited ditto, recrystallized < 0.001% Co.001% Co.001% Fe, < 0.001% Co.001% Co.001% Co.001% Co.001% Co.001% Co.0009% C, trace Ag, bal. Ni	Fetz	1935	

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0.11% Co, 0.013% Fe, trace Si + S. dieterolytic Ni, as deposited ditto, recrystallized <0.001% Cu, <0.001% Cu, <0.001% Fe, <0.001% Cu, 0.0001% Fe, <0.001% Co, 0.0000% C, trace Ag, bal. Ni =

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ingly low recovery point at $T_r = 410$ degrees Cent. is shown by Ransley and Smithells²² although they used nickel of only 99.62 per cent purity. Apart from clarifying disagreement in the literature, a new investigation as to whether highly pure nickel softens at a temperature still lower than found by Ransley and Smithells offered interesting possibilities.

Previous Recovery Tests with Cold-Worked Nickel

The changes in hardness and tensile strength of cold-rolled nickel after annealing at various temperatures are shown in Fig. 1 while Fig. 2 refers to nickel reduced by methods other than rolling. The experiments of Ransley and Smithells are given separately in Fig. 3.

Apparently the first crude test on the softening of cold-worked nickel was carried out by Matweef²⁸ who hammered 25-centime coins, and heated the samples for 5 minutes at 600, 800 and 1150 degrees Cent. In spite of the limited number of tests, it is evident from curve a, Fig. 2, that pronounced softening occurs around 600 degrees Including in this chronological review the experiments of Rose,²⁴ we must be reminded that the rebound hardness unlike penetration hardness is strongly affected by the elastic properties of the testing material.25 Evaluation of the "hardness" curve b, Fig. 1, of Rose yields $T_r = 550$ degrees Cent.—Guillet²⁶ gives no information on reduction and purity of the nickel he used. The mean value of two recovery curves, curve c, Fig. 2, shows T_r = 690 degrees Cent.— Price and Davidson²⁷ utilized relatively impure nickel, cold-rolled it 73 per cent, heated it up to temperature in 25 minutes, and held it there for 30 minutes. Deviations of 40 kilograms per square millimeter (56,880 pounds per square inch) between the hardness results under 500 and 3000 kilogram loads occur. Curve d, Fig. 1, giving tensile strength determinations, shows the pronounced softening Tr 650 degrees Cent.—Schottky and Jungbluth²⁸ used rather impure nickel upset 5-50 per cent. The hardness values fluctuated considerably and depended greatly upon the section of the sample tested ow-

²²C. E. Ransley and C. J. Smithells, Journal, Institute of Metals, Vol. 49, 1932, p. 287.

²³M. Matweef, Revue de Métallurgie, Vol. 8, 1911, p. 708.

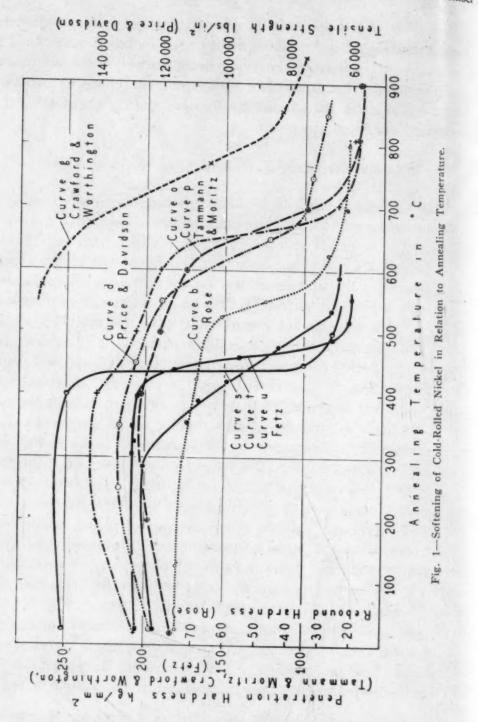
²⁴T. K. Rose, Journal, Institute of Me. 's, Vol. 8, 1912, p. 86.

³⁵E. Fetz, *Proceedings*, American Society for Testing Materials, Vol. 35, II, 1935, p. 318 (Discussion).

²⁸L. Guillet, Revue de Métallurgie, Vol. 10, 1913, p. 665.

²⁷W. B. Price and P. Davidson, Transactions, American Institute of Mining and Metallurgical Engineers, Vol. 64, 1920, p. 414.

²⁸H. Schottky and H. Jungbluth, Kruppsche Monatsh, Vol. 4, 1923, p. 197.



ing to the method of cold-working. The recovery curve e, Fig. 2, refers to a specimen upset 50 per cent. The "beginning of pronounced softening" may be placed at 400 degrees Cent. and T_r at 510 degrees Cent. whereas recrystallization started at 550 degrees

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Cent.—An evaluation of the hardness curve f, Fig. 2, by Mochel²⁹ vields T_r = 590 degrees Cent.—Results published by Crawford and Worthington³⁰ can hardly be used for comparison, as all samples were heated first to 578 degrees Cent. and then brought up in 1 hour to higher annealing temperatures. The recovery curve g, Fig. 1, of a sample cold-rolled 50 per cent thus shifted to 740 degrees Cent. One set of samples annealed directly at 500 degrees Cent. for 1000 hours shows partial softening of the more severely worked specimens. (Table II.) The hardness increase of the slightly deformed sample is significant.—Nickel drawn 23 and 53 per cent softens at $T_r = 720$ degrees Cent. and 640 degrees Cent. (curve h, Fig. 2) respectively, according to Ancelle.31 Hardness maxima between 300 and 350 degrees Cent. are correlated with the magnetic transformation. Recrystallization was observed at a higher temperature than softening. A wire of 99.5 per cent pure (?) nickel was found by Kawai¹⁷ to recover from a 31 per cent reduction at 650 degrees Cent. (curve i, Fig. 2)— Ransley and Smithells²² annealed nickel wires of commercial purity (curve j, Fig. 3), and of refined nickel with and without (curve k) additions of 0.34 manganese, (curve 1), 2.33 per cent iron (curve m), and 0.07 per cent magnesium (curve n) for 5 minutes in hydrogen at various temperatures. Whereas 0.07 per cent magnesium, which metal is widely used as deoxidizer for nickel, shifts the recovery temperature of 99.62 per cent nickel from 410 degrees Cent. to about 620 degrees Cent., 2.33 per cent iron raises T_r to only 550 degrees Cent. Recrystallization occurred always at higher temperatures than softening. (Table III.) Refined nickel recrystallized at 480 degrees Cent., the lowest temperature recorded for nickel so far. Curve o and p, Fig. 1, of Tammann and Moritz¹² shows that 60 and 90 per cent reduced nickel recovers respectively at $T_r = 670$ and 660 degrees Cent. An evaluation of the recovery test of Wilson and Thomassen³² with 99.8 per cent pure nickel, upset 60 per cent yields $T_r = 580$ degrees Cent. (curve q, Fig. 2). The latest softening curve r, Fig. 2, of Ellis³³ shows an unusually high value for $T_r = 800$ degrees Cent.

The loss of strength at elevated temperatures of cold-worked

²⁹N. L. Mochel, *Proceedings*, American Society for Testing Materials, Vol. 28, 1928, II, p. 292.

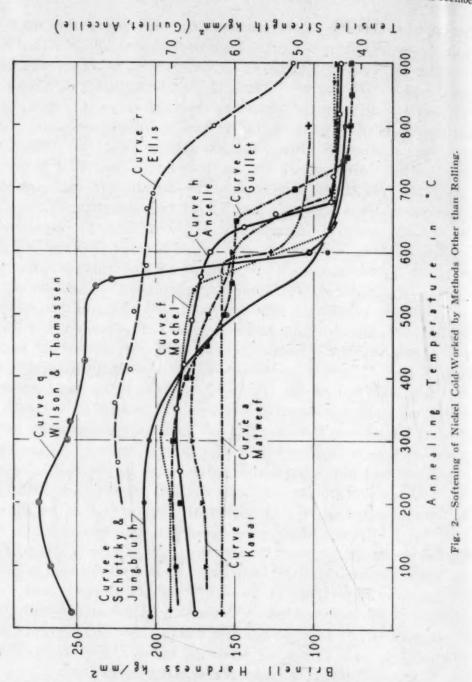
⁸⁰C. A. Crawford and R. Worthington, "Symposium on Effect of Temperature on the Properties of Metals." American Society of Mechanical Engineers and American Society for Testing Materials, 1931, p. 557.

³¹A. Ancelle, Revne de Métallurgie, Vol. 30, 1933, p. 266.

³²J. E. Wilson and L. Thomassen, Transactions, American Society for Metals, Vol. 22, 1934, p. 19.

³³O. W. Ellis, Journal, Institute of Metals, Vol. 54, 1934, p. 145.

Reduction in Per Cent



nickel has also been investigated.^{22, 34, 35, 36} The beginning of marked softening can be determined rather safely and in agreement with room temperature tests. However, at relatively high temperatures, pulling speed becomes a factor, and the recovery curves do not take

³⁴W. P. Sykes, *Transactions*, American Institute of Mining and Metallurgical Engineers, Vol. 64, 1920, p. 780.

asD. H. Ingall, Journal, Institute of Metals, Vol. 32, 1924, p. 41.

³⁶H. Schaarwächter, Nickelhandbuch, Sect. "Nickel," p. 23.

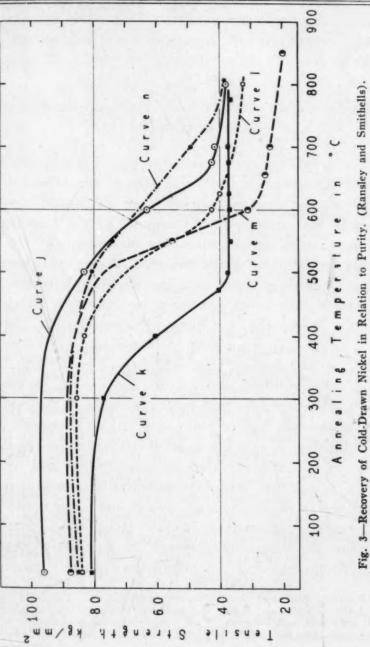
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Table II Softening of Nickel After a 100-Hour Anneal at 500 Degrees in Relation to Reduction²⁰

Reduction		Brinell Hardne	ess in kg/mm ² †	Hardness Change in Per Cent
in Per Cent		Before Annealing	After Annealing	with Reference to Annealed State*
4.4		156	170	+12
15	171	195	196	* *
31		235 264	235	
46	314	264	235	-28
59		288	183	-56
80		320	136	-84

†Converted Rockwell Numbers. *Annealed: 57 Rockwell "B" = 100 Brinell.



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Table III
Softening and Recrystallization of Nickel in Relation to Purity

			Refine	d Nickel	
Material	Commercial Nickel	Without Addition	+ 0.07% Mg	+ 2.33% Fe	+ 0.34% M
Recrystallization					, , , , , , , , , ,
Temperature in Degrees Cent. Recovery Tem-	640	480	640	580	. 620
perature Tr of Tensile Strength	600	410	620 -	550	550

a horizontal course after the release of strain hardening thus rendering the location of T_r impossible.

Re-Determination of the Hardness Recovery of Cold-Rolled Nickel

Nickel refined by several redepositions and containing 0.11 per cent cobalt, 0.013 per cent iron and traces of silicon and sulphur, was cold-rolled 80 per cent. Samples were suspended directly on a thermocouple hot-junction in a furnace of large heat capacity and annealed at various temperatures for 30 minutes. The annealing temperature was attained in 2-3 minutes. After quenching in water, the hardness was determined with the Rockwell hardness tester and the hardness values were converted to the more commonly used Brinell units using the formulas of Petrenko.³⁷ The hardness changes in relation to temperature are presented in curve s, Fig. 1, yielding T_r = 440 degrees Cent. It seemed possible that this relatively low value resulted partly from the use of electro-deposited nickel. The comparatively high initial hardness of the rolled samples of 250 kilograms per square millimeter (355,500 pounds per square inch) is indicative, as it is about 30-40 Brinell numbers higher than the maximum hardness reported on cold-worked and recrystallized material. Furthermore, hardness anomalies have been noted on electrolytically deposited nickel, iron, cobalt and chromium. Guichard and co-workers88 prepared electrolytic nickel whose hardness was 365 kilograms per square millimeter. The extremely great hardness of electrolytic chromium is generally known; Kroll³⁹ recently proved 1937

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 ³⁷S. N. Petrenko, Bureau of Standards, Journal of Research, Vol. 5, 1930, p. 19.
 ³⁸M. Guichard, Clausmann, Billon and Lanthony, Bulletin, Soc. Chim. France, (5)
 Vol. 1, 1934, p. 679.

²⁰W. Kroll, Z. anorg. und allg. Chem., Vol. 226, 1935, p. 23.

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that the hardness of chromium is only 150 Brinell. In some cases extremely great hardnesses have been attributed erroneously to the presence of hydrogen.40 The hydrogen content of 10 different electrolytic nickel preparations, however, did not disclose any interrelation between these two factors, and unequivocal proof was furnished

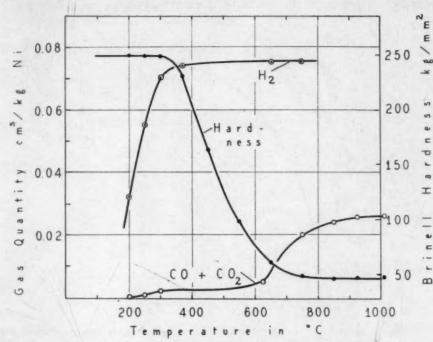


Fig. 4-Hardness Changes and Gas Liberation of Electrolytic Nickel. (Guichard and Co-workers).

on annealing: hydrogen was completely liberated before the hardness began to drop. (Fig. 4). Neither can the CO and CO2 contents be blamed for the great hardness. The hardness-temperature curve shows a T_r value of about 460 degrees Cent., surprisingly close to the T_r value for cold-rolled electrolytic nickel itself. The explanation that the hardness is due to ultra-fine grain size agrees with X-ray findings of Wood⁴¹ who proved with electrolytic nickel that the diffusion of the X-ray diffraction spectra must be attributed to finegrained structure, though additional lattice stresses might occur. Makariewa and Birükoff⁴² confirmed with electrolytic chromium that there is no interrelation between hardness and hydrogen content, but between elastic lattice stresses, recrystallization and hardness. Unfortunately, the microstructures shown by the French investigators³⁸

⁴⁰W. Köster, Z. Metallk., Vol. 20, 1928, p. 189.

⁴¹W. A. Wood, *Philosophical Magazine*, (7), Vol. 15, 1933, p. 553. ⁴²S. P. Makariewa and N. D. Birükoff, *Z. Elektrochem.*, Vol. 41, 1935, p. 623.

include no information on the heat treatment of the recrystallized electrolytic nickel to decide the question whether electrolytic nickel begins to soften at 310 degrees Cent. due to release of internal stresses or to recrystallization.

Since Köster⁴⁰ established that cold-rolled ordinary copper, electrolytic copper and recrystallized electrolytic copper recover from

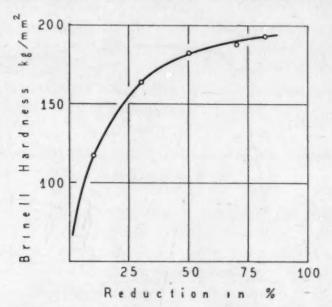


Fig. 5-Strain Hardening of Recrystallized Electrolytic Nickel During Cold Rolling.

cold work in a greatly different manner, the experiments described above were repeated with recrystallized material. The electrolytic nickel was annealed in hydrogen for 1 hour at 1000 degrees Cent., cold-rolled 20 per cent, annealed for 2 hours in a vacuum at 700 degrees Cent. and finally cold-rolled from 10 per cent to 82 per cent. Greater reductions were prohibitive as the hardness determinations became unreliable. A confirmation of Moore's⁴³ observations that an abrupt increase of hardness takes place at reductions exceeding 98 per cent would be of interest and would indicate that the individual atoms might be affected to a greater extent by extreme reductions.

The progressive strain hardening of recrystallized electrolytic nickel in relation to cold-rolling is shown in Fig. 5, the considerable increase of hardness in the initial stages being remarkable. At about 15 per cent reduction, the maximum gain of hardness has been attained. Although Schmid and Müller⁴⁴ found that the broadening

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⁴³H. Moore, Journal, Institute of Metals, Vol. 32, 1924, p. 407.

⁴⁴W. Schmid and A. W. Müller, Z. techn. Physik, Vol. 16, 1935, p. 161.

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of X-ray lines increases linearly with rising tensile stresses, Wood⁴⁵ observed that lattice changes of metals—when they occur at all—increase up to only 25 per cent deformation and that at 35-50 per cent reduction preferred orientation begins. The broadening of the in-

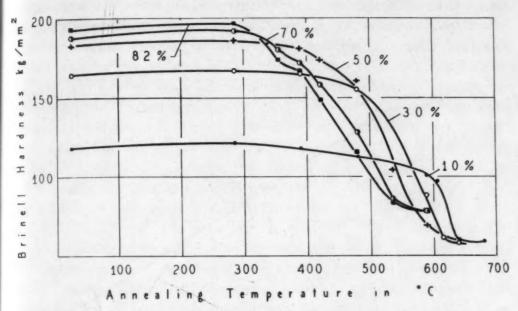


Fig. 6-Softening of Cold-Rolled, Recrystallized Electrolytic Nickel in Relation to Reduction.

terference lines of nickel yielded the following values in relation to cold-rolling:

Percentage reduction 3.9 12 22 29 37 48 58 68 73 Line width in mm. 0.29 0.57 0.86 1.14 1.14 1.00 1.14 1.00 1.00

The recovery of nickel from various reductions is shown in Fig. 6 which confirms practical experience that the ease with which energy stored in cold-worked metal is released by heating is proportional to the amount of reduction. The recovery from cold work of the 82 per cent reduced sample, expressed by curve t, Fig. 1, proves that T_r is shifted to slightly higher temperatures by the recrystallization and that the softening range is widened up. Contrary to the uneven strain hardening with increasing cold work, the T_r -values of the individual recovery curves are a straight line function of the reduction. (Fig. 7.) 99.866 per cent nickel recovers from a reduction of 5-95 per cent in an interval of 410-620 degrees Cent. The conclusion of Tammann and Moritz¹¹ that "the temperature range in which hard-

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⁴⁵W. A. Wood, Philosophical Magazine, (7), Vol. 14, 1932, p. 656.

ness recovers depends but little on the amount of cold work" could not be verified. Nickel cold-worked 90 per cent recovers at a 70 degrees Cent. lower temperature than 60 per cent reduced nickel rather than at a 10 degrees Cent. lower temperature. Although the hardness was found to recover at a temperature 200 degrees Cent. lower than given by Tammann and Moritz, their statement still holds true that electric conductivity and hardness recover in two separate temperature

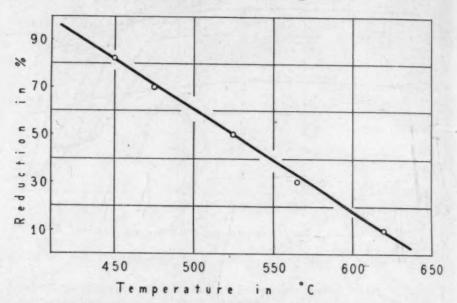


Fig. 7-Relationship Between Recovery Temperatures Tr and Reduction of Nickel.

ranges and that these two physical properties are apparently affected by different phenomena during cold work and subsequent annealing. The statement of Tammann and Rocha¹⁵ that the recovery of magnetic induction and hardness coincide, in contradistinction to iron, needs confirmation.

The slope of the curve in Fig. 6 has probably some physical significance. It would be of interest to learn whether the same curve will be obtained with (1) all metals belonging to the face-centered system, (2) with reference to different cold-working methods and degrees of purity of testing materials, and (3) with reference to other physical properties affected by cold work. Tammann and Bandel,¹⁴ for instance, found that the e.m.f. of 99.999 per cent silver recovers linearly from a reduction of 10-90 per cent in a 210 degrees Cent. temperature range.

Although the nickel employed above is one of the purest grades of nickel so far used for recovery experiments, the test has been re1937

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peated with probably the purest nickel ever prepared. Spectroscopic analysis revealed the presence of copper, cobalt, iron and a trace of silver. Quantitative chemical analysis showed that each of the metals copper, cobalt and iron were present in quantities less than 0.001 per cent and that the carbon content was 0.009 per cent. This metal, prepared by electrolysis of Kahlbaum's NiCl₂ is, therefore, almost 99.99 per cent pure. Powder scraped from the cathode was compressed under high hydraulic pressure and heated in a vacuum close to the melting point of nickel. Contamination from the highly refractory alundum crucible was practically excluded by the use of the high frequency induction heating. The slug was then cold-rolled 30 per cent and again exposed to about 1350 degrees Cent. in a vacuum. After each vacuum treatment, the surface of the metal was ground off. Finally the sample was cold-rolled 80 per cent and samples were heated for 30 minutes at various temperatures. The computed Brinell hardness values are presented in curve u, Fig. 1, which yields $T_r = 463$ degrees Cent. The difference between the recovery of the highly pure and the 99.866 per cent pure nickel is surprisingly small. Apparently cobalt, so closely related to nickel, does not affect the softening of nickel in quantities as high as 0.11 per cent. It is worthy of note that the precision lattice constant determination led to the same value for both materials and agrees with redeterminations by Jette and Foote⁴⁶ on the same materials: $a = 3.51684 \pm 0.00008$ A. at 25 degrees Cent.

DEBYE LINES AND STRAIN HARDENING EFFECTS

The findings of Wilson and Thomassen³² regarding the sharpness of the X-ray spectra of cold-worked nickel after annealing at various temperatures, namely that the Debye lines become sharp at a lower temperature than the loss of hardness, are qualitatively in agreement with the author's. X-ray pictures were taken on the same material represented in curve s, Fig. 1. The doublets of the interference lines with the largest reflection angles (220- and 311-planes) became as sharp as in fully annealed nickel at a temperature where the drop in hardness amounted to only 20 per cent. Apparently a distinction must be made between the lattice distortions produced by cold-working and the hardness recovery, just as the strain hardening effects increase beyond 25 per cent reductions without further widen-

⁴⁶E. R. Jette and F. Foote, Journal of Chemical Physics, Vol. 3, 1935, p. 605.

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ing of the X-ray lines. A redetermination of the lattice parameter after the diffraction lines became sharp has not yet been carried out. It appears logical that the latent energy remaining after cold work might widen up the space lattice as is the case with rising temperatures due to an increase of the total lattice energy. Wood⁴⁷ attempted to compute the energy stored in cold-worked copper from the broadening and shifting of the interference lines and by a direct comparison with the same effect brought about by heating.

Experiments by Dehlinger⁴⁸ prove that the broadening of Debye lines need not necessarily accompany cold hardening. Severely cold-rolled aluminum shows distinctly split Kα-doublets, while tantalum, which strain hardens but little after a 5 per cent reduction, furnishes completely diffuse lines. Wood,⁴⁹ however, pointed out that in the case of cold-worked aluminum and nitrided steel, the hardness increase manifests itself in a fading away of the high order spectra due to a derangement of the electron distribution about the atoms.⁵⁰ Room temperatures appear to be an arbitrary reference point since a broadening of the X-ray lines takes place when aluminum is cold-worked 60 per cent at −76 degrees Cent.³²

In this connection, attention may be directed to a further hardness anomaly observed with synthetic metallic bodies of highly disperse powders pressed at elevated temperatures. Trzebiatowsky⁵² attained hardness values as high as 190 Brinell with copper powder although the maximum hardness of ordinary rolled copper is 110-130⁴³ Brinell. The former phenomenon is attributed to peculiarities of the method of cold work and to the high degree of dispersity of the crystals. Trzebiatowsky observed all the characteristic features that accompany the strain hardening of regular metals, such as lack of Arkel split-up, broadening of interference lines, abnormal decrease of the effective scattering power of atoms at high angles. But it is highly significant that the Arkel lines are sharp at 350 degrees Cent., while the hardness is still over 100 Brinell at 600 degrees Cent.! A remarkable residual hardness remained which is not revealed by the X-ray picture as lattice distortions.

⁴⁷W. A. Wood, Philosophical Magazine, (7), Vol. 18, 1934, p. 495.

⁴⁸U. Dehlinger, Z. Krist., Vol. 65, 1927, p. 615.

⁴⁹W. A. Wood, Philosophical Magazine, (7), Vol. 16, 1933, p. 719.

⁶⁰R. Brill, Z. Physik, Vol. 61, 1930, p. 454.

⁶¹ J. Hengstenberg and H. Mark, Z. Physik, Vol. 61, 1930, p. 435.

⁸⁸W. Trzebiatowsky, Z. phys. Chem., (B) Vol. 24, 1934, p. 75; (A) Vol. 169, 1934, p. 91.

DISCUSSION OF THE HARDNESS RECOVERY EXPERIMENTS ON NICKEL

As was to be expected, the curve of the T_r values plotted against composition for the binary copper-nickel system is of the same type as that of electrical resistance and alloy hardness, in binary systems built up by a series of solid solutions with random atom distribution.

In qualitative agreement with the determinations of Ransley and Smithells,22 the softening of highly pure, cold-worked nickel was found to take place at considerably lower temperatures than have been established previously. But in spite of the higher purity of the author's nickel, the T_r-value of the hardness recovery curve of recrystallized nickel cold-rolled 70 per cent is about 70 degrees Cent. higher than the corresponding value on the tensile strength recovery curve of a nickel wire drawn 70 per cent. It appears improbable that this discrepancy is due to the different physical property determined.²⁰ Simultaneous determination of the recovery of tensile strength and hardness led to substantially the same recovery temperature range. It is possible that the divergency is due to the different methods of cold-working, viz. rolling versus drawing. Energy tests by Taylor and Quinney⁵³ support this assumption. They proved that 100 times as much latent energy may be stored in copper by twisting as by tension. Compression was still more effective than torsion. The percentage reduction may also be too arbitrary a basis for different working methods and might better be replaced by actual strain hardening effects. A tensile strength of 81 kilograms per square millimeter (115,106 pounds per square inch) of the refined nickel sample drawn 70 per cent would correspond to a Brinell hardness of 235 kilograms per square millimeter (334,170 pounds per square inch),33 i.e., to a strain hardening not attained in the rolling tests of the author. A distinct difference between the recovery of nickel wires all drawn 75 per cent but drawn to various diameters can be observed in the experiments of Ransley and Smithells.²² Tammann and Bandel¹⁴ point out that drawing exerts a considerable influence upon the e.m.f. of nickel while torsion is almost without any effect.

The hardness recovery curve k, Fig. 3, of Ransley and Smithells²² shows that softening of highly pure nickel begins below 200 degrees Cent. This observation could not be verified as shown by the following mean values of 4-6 determinations after 30 minutes annealing:

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⁵³G. I. Taylor and H. Quinney, *Proceedings*, Royal Society, London, Vol. 143, 1934, p. 307.

Brinell hardness before annealing	205.3 207.8	325 203.0 206.8 3.8	350 202.5 207.5
Hardness increase	2.5	3.8	5.0

The 99.866 per cent nickel samples partly softened at 300 degrees Cent., but there is an increase of hardness at 287 degrees Cent. in relation to reduction as follows:

Reduction in per cent	10	30	50	70	82
Brinell hardness before annealing	118	164	183		193
Brinell hardness after annealing	121	167	186	192	197

No additional determinations were made by Ransley and Smithells22 below 400 degrees Cent., a particularly unfortunate omission in view of the very small but distinctive inflection exhibited by their tensile strength curve at elevated temperatures. The long arrests on other tensile strength/temperature curves taken at elevated temperatures, however, support the assumption that the contaminations have something to do with that increase of strength before softening begins. This hardness anomaly was observed by many investigators and, if mentioned at all, was generally connected with the magnetic transformation point of nickel. This interpretation appears to be dubious, because this phenomenon occurs in a wider temperature range than could possibly be associated with the effect of contaminations upon the Curie point and because it occurs also in nickelsilver,27,54 semi-hard and hard carbon steel,31,55 nickel steel,28 electrolytic iron,56 and brass.57,58 Inasmuch as this increase occurs in annealed nickel, it cannot be ascribed to cold-working. The present author assumes that this phenomenon is connected with the minute effect which precedes the Curie point,59 and which can also be observed in alloys undergoing a transformation in a temperature interval.

CRITICAL DISCUSSION OF INVESTIGATIONS ON THE RECOVERY OF METALLIC MATERIALS FROM COLD-WORKING

A critical glance over the recovery data on nickel presented in Table I and Figs. 1 to 3 raises the following questions: (1) What

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⁵⁴F. C. Thompson, Transactions, Faraday Society, Vol. 12, 1917, p. 32.

⁶⁶M. Grenet, Revue de Métallurgie, Vol. 6, 1909, p. 1054.

⁶⁶F. Oberhoffer and W. Oertel, Stahl und Eisen, Vol. 39, 1919, p. 1061.

D. Bengough and O. F. Hudson, Journal, Institute of Metals, Vol. 4, 1910, p. 92.
 H. Bassett and C. H. Davis, Transactions, American Institute of Mining and Metallurgical Engineers, Metals Division, 55, 1928.

⁵⁹ A. Schulze, Phys. Z., 37, 41, 1936.

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is responsible for the great deviations of 400 degrees Cent. between the T_r-values and (2) what refinements of our present experimental technique are imperative?

Besides the amount of cold work, the chemical analysis exerts the greatest effect upon the softening of cold-worked nickel. The individual contaminations affect the hardness recovery to a varying degree and in an unknown manner. Even if one takes into account atomic instead of weight percentages, it is surprising that for instance, 0.07 per cent magnesium already raises the softening temperature of pure nickel by 210 degrees Cent. and 2.33 per cent iron only by 140 degrees Cent. A redetermination of the softening of highly pure metals and systematic, quantitative research on the effect of the various elements added individually and in combination to chemically pure metals, and a determination of the lower critical concentrations of impurities appear to be urgent desiderata. In this respect, the added elements must be kept in solid solution at all temperatures, otherwise precipitation hardening effects would obliterate the release of strain hardening. Because the quantitative chemico-analytical determination of impurities is often difficult, spectroscopic analysis is an attractive possibility. At all events it is essential that investigators state the purity and history of their testing material. Several analyses in Table I are of questionable value for it is inexcusable to determine analytically only those elements present in larger quantities, to pass the balance as nickel plus cobalt, and to neglect the "less important" contaminations the sum of which may decidedly affect the recovery from cold work.

Statements on the reduction are missing in several papers. The significance of this factor is clearly brought out by the present determinations, showing that softening is lowered by 23.3 degrees Cent. with every 10 per cent increase of reduction. A comparative investigation of the recovery of nickel cold-worked by different methods in relation to amount of cold work or to strain hardening is still lacking. It is advisable at present to standardize on either rolling or drawing as a method to secure comparable results.

It seems to have become a standard practice to apply annealings of 30 minutes duration. Many investigations of the effect of annealing time on the softening of cold-worked materials have been carried out. Recent tests of the author²¹ on cold-worked copper, copper-tin and copper-nickel alloys of technical purity, for instance, proved that longer annealing periods may result in a partial softening

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at lower temperatures. However, a characteristic point can hardly be chosen on hardness/annealing time curves, so that a standardized 30-minute heat treatment is most advisable. No attention has been paid so far to the time for bringing the sample to the annealing temperature. This should be done as quickly as possible in view of the following observations of the author. If one anneals a cold-worked copper-nickel sample slightly below that temperature at which the first indications of a loss of strength appear and then holds the specimen for 30 minutes at a higher annealing temperature, the loss of hardness will be less pronounced than with a sample cold-worked to the same extent and brought up directly to the higher annealing temperature. Evidently the former sample loses part of its latent energy induced by cold work so that it now corresponds to a less deformed sample and consequently recovers at a higher temperature.

Recovery of cold-worked purest metals is not only of scientific interest but also of considerable technological importance, which will certainly increase progressively. A comparison between the former and present analyses of commercial nickel, copper, cadmium, iron, aluminum, magnesium, etc., strikingly demonstrates the remarkable technical advances in the production of metals of high purity. The practical application of recovery tests with highly refined metals is clearly brought out by the following typical cases. Sheets of pure copper completely soften on tinning in a 50/50 tin-lead bath of 360 degrees Cent. By adding only 0.034 per cent silver this is prevented due to raising the recovery curve by 130 degrees Cent. Cold-worked copper recovers only partly from a 38 per cent reduction at 400 degrees Cent. owing to the presence of 0.4 per cent antimony.61 Recrystallization of chemically pure silver takes place at room temperature; 0.055 per cent iron lowers the recrystallization of pure silver from 150 to 20 degrees Cent., and cold-worked silver becomes brittle after a short time due to contaminations with iron. 62

Systematic research on the phenomenal effect of impurities on the recovery of cold-worked, highly pure metals should yield some clues of the nature of strain hardening in particular, and perhaps on the nature of hardness in general.

⁶⁰ Unpublished experiments.

eiS. L. Archbutt and W. E. Prytherch, Journal, Institute of Metals, Vol. 45, 1931, p. 265.

⁶²H. Widmann, Z. Physik, Vol. 45, 1927, p. 200.

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SUMMARY

(1) The turning point of the hardness recovery curve of highly pure (99.99 per cent) 80 per cent cold-rolled nickel was found at T_r = 463 degrees Cent. (30-minute anneal).

(2) Electrolytic, 99.866 per cent nickel, as deposited, recovers from an 80 per cent reduction at $T_r = 440$ degrees Cent. In recrystallized material, T_r shifted to 454 degrees Cent.

(3) The turning points of the hardness recovery curves of cold-rolled nickel are directly proportional to the amount of cold work.

(4) Increasing the reduction by 10 per cent lowers the softening temperature by 23.3 degrees Cent.

(5) Contrary to the marked effect of other contaminations present in commercial nickel, 0.11 per cent cobalt did not affect the recovery temperatures.

(6) In the binary system nickel-copper, additions of nickel to copper and of copper to nickel increase the softening temperature of cold-worked material.

(7) The increase of strain hardening during cold-rolling of nickel is not a linear function of the amount of cold work.

(8) In cold-rolled electrolytic nickel, the Debye lines become completely sharp on annealing before the hardness recovers from cold work.

(9) A slight increase of hardness takes place before the release of strain hardening. This maximum of hardness has nothing to do with the Curie point of nickel. It might be connected with the slight rearrangement of atoms preceding or initiating the magnetic transformation.

(10) Determinations of the recovery of tensile strength and hardness of nickel from cold work are historically reviewed.

(11) Refinements of our present experimental technique for investigation of the recovery of physical properties of metallic materials from cold work are critically discussed.

ACKNOWLEDGMENT

The author wishes to express his indebtedness to Dr. W. A. Wesley of the International Nickel Co., Research Laboratory, Bayonne, N. J., for placing at his disposal some highly pure nickel and analytical data.

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DISCUSSION

Written Discussion: By E. P. Polushkin, consulting metallurgical engineer, 500 West 111th Street, New York City.

Recovery of cold-worked metals on annealing is one of the most difficult problems in physical metallurgy, and the author should be congratulated with the thorough scientific manner in which he treats this problem. I would like to comment on the possible reasons for a sharp disagreement of various investigators in regard to the softening temperature of cold-worked nickel, as reported in Table I of this paper. It seems to me that this temperature depends not only on the purity of the metal and on the amount of cold work applied. but also on the specific kind of cold work. The latter factor should not be overlooked. In general, the behavior of metals in cold-working processes is greatly influenced by the nature of the deforming stresses and their distribution within the metal. A remarkable illustration of this effect is found in low carbon steel tubes. When they are reduced by the ordinary bench drawing process, the maximum reduction permissible without annealing is 30 to 40 per cent, while by applying Rockrite process the reduction of same tubes can be carried out almost indefinitely. In the first process mostly tensional stresses are employed and in the second mostly compressive stresses. Recrystallization and recovery of the physical properties also depend on the specific kind of cold work. In our annealing experiments on low carbon steel tubes, two series of specimens were prepared, both cut from the same original hot-rolled tube, but reduced by two different processes, and in one series the temperature of recrystallization was found to be from 25 to 50 degrees higher than in the other series. I believe that recovery of physical properties in these tubes would also occur at different temperatures. The amount of reduction is not yet a criterion of the inner condition of a cold-worked metal, for in two similar pieces with equal reduction the deformation of grains may differ to such an extent that one metal may be close to the breaking point while the other may be capable of consuming a large amount of further work without failure. Distribution of stresses within the metal is likewise of a great importance. In fluids the stresses are distributed uniformly, but in solid materials their magnitude varies from point to point, and so does the degree of plastic deformation. Strained metals, therefore, are not homogeneous materials, and in all our tests a possible variation of the local conditions should not be disregarded.

There is another point of interest that I would like to touch upon. The author points to a large gap between the temperatures of softening and recrystal-lization in certain metals, and concludes therefrom that no close relation between these two phenomena exists. Of course, recrystallization is a continuous process within a certain range of temperatures. A definite temperature usually assigned to it marks only one of its final stages when all grains of the original structure disappear. Incipient recrystallization starts much earlier, with the first visible changes in the slip markings and in the boundaries of grains which become serrated lines. On the other hand, parallel alignment of grains caused by cold work can still be found in metal annealed far above its conventional recrystallization temperature. With the broad conception of recrystallization process, it is natural to expect that restoration of physical

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Written Discussion: By J. E. Wilson, metallurgist, Bausch and Lomb
Optical Company, Rochester, N. Y.

I have read Mr. Fetz's paper with a great deal of interest, and I believe he is to be complimented on a careful piece of experimental work. In order to determine what impurities might cause the higher recovery temperature of nickel used in the experiments reported by Prof. Thomassen and myself¹ the impurities in our sample were checked by means of a large Littrow spectrograph. Small amounts of cobalt, manganese, magnesium, and silicon were found, with faint traces of aluminum, iron and silver. From the data presented in the paper, it would seem that the presence of magnesium in our nickel was effective in increasing the recovery temperature.

With regard to Mr. Fetz's explanation of the increased hardness of the electrodeposited and untreated nickel as compared to other specimens after equivalent degrees of cold work, it is notable that not only is the hardness considerably greater but the temperature range of recovery is smaller. would indicate a much narrower distribution of energy in the untreated, electrodeposited nickel. Can this phenomenon be entirely explained by the fine grain size hypothesis? H. N. Huntzicker and L. Kahlenberg² found that if a piece of soft nickel foil be made cathode in a H₂PO₄ bath and hydrogen evolved on it for 30 minutes, a great increase in hardness will result. In the words of these writers, the foil "becomes so hard that it breaks off sharply on being bent." Other effects of hydrogen on nickel are to increase its activity greatly as measured by its power to reduce salt solution; the nickel, conversely, has equally remarkable effects on the hydrogen evolved, causing it to become "activated" in a special way. In view of such evidence, it seems fairly clear that hydrogen, absorbed by the nickel either by heating in its atmosphere or by electrolytic action, has of itself definite effects on the physical properties of nickel. I do not think we have sufficient experimental evidence at present to conjecture mechanism of these phenomena, but I do not feel we are justified in ignoring their existence. In connection with Mr. Fetz's qualitative confirmation of our findings that line sharpening occurs at a somewhat lower temperature than the recovery in hardness, I believe that it is essentially a part of the recovery phenomena that the atoms diffuse back to their proper lattice points before the remaining stored up energy may be released.

Oral Discussion

M. J. Donachie: I have found Mr. Fetz's paper very interesting, having noticed a somewhat similar phenomena in a solid solution type of alloy. Specifically the alloy is a phosphor bronze alloy, approximately 92.1 per cent copper, 7.5 per cent tin, 0.4 per cent phosphorus with iron as an occurring impurity. I wish to state that this was not a scientific investigation to determine the recovery points of the alloy.

² Recovery of Metals and Alloys from X-Ray Line Broadness and Its Relation to High Temperature Stability," J. E. Wilson and L. Thomassen, Transactions, American Society for Metals, September 1934.

[®]Transactions, Electrochemical Society, Vol. LXIII, 1933, p. 349.

³Engineer, Alloy Department, George W. Prentiss & Co., Holyoke, Mass.

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In the demands placed on this alloy it is necessary to produce a wide range of physical properties. Many of these physical properties are obtained in structures below the normal full annealed crystalline state. The tolerance on any individual set of physical properties is very narrow. If definite properties were to be had and controlled in the smaller grain size range, it was essential that the effect of cold-working and annealing be investigated and the structural changes followed with the microscope.

The findings showed that a preferred cold-working and annealing were required to hold certain physical properties within desired limits. One of the things revealed was an annealed structure observed and recorded under the microscope as being of extreme fineness. This structure did not show a definite polyhedral boundary up to 500 diameter magnifications. There were many fine lines present that might be attributed to the effect of previous cold-working. The outstanding feature is that the physical properties are comparable with those in the fully annealed condition of normal grain size. I have formed no definite opinion as to the exact condition of the structure. The thought occurred that it may be the first stage of incipient recrystallization and it has considerable importance attached to it in our work. There was no critical relation observed between the cold work and the temperature of normal full annealing. It was found, however, that a relationship did exist between cold work and softening temperature.

I now wish to ask Mr. Fetz a few questions. I assume that in the work described, the cold-rolling was from the ingot. First—if there were any traces of residual cold work existing after the heat treatment. Was there any cold work done following a heat treatment; that is, extended cold-working, including rolling, annealing and further rolling? Does this cause a shift in the recovery or restoration curve? Were there any microscopic observations made, and if so, what kind of structures were obtained?

Author's Closure

I fully agree with Mr. Polushkin that the distribution of the deforming stresses profoundly affects the behavior of metals in cold-working and recovery therefrom. I have noticed on a number of samples that even the upper and lower sides, in spite of their relative proximity, do not soften in exactly the same temperature range; I have also suggested that the difference in the coldworking methods (drawing versus rolling) may be responsible for the different Trivalues obtained by Ransley and Smithells and by me.

It is interesting to learn that a process has been developed in which "the reduction of low carbon steel tubes can be carried out almost indefinitely." To date, gold and silver, highly pure copper and aluminum have been outstanding in their defiance of all laws of plastic deformation, as they do not require intermediary annealings. It would be interesting to learn whether the strain hardening effects in the Rockrite process are lower than in customary bench drawing. I do believe that this is the case, since the amount of reduction is not necessarily a criterion of the latent energy stored by cold-working; the latter depends also on the deforming tools, lubricants, initial cross sectional area, speed of plastic deformation, etc.

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ch "the mitely." en outdo not her the stomary reducorking; ectional The recrystallization temperature is no physical constant. We know only that it is approximately inversely proportional to the logarithm of the annealing time. Mr. Polushkin's many years of experience with the microscope, particularly with the art of high magnification microscopy, naturally have given him abundant opportunity to observe details during recrystallization which escaped the attention of most of us research metallurgists. Whereas the recovery of such physical properties as electrical or thermal conductivity, hardness, tensile strength, etc., can be accurately measured with negligible "personal error," no scientific definition of the "recrystallization temperature" exists. Obviously the establishment of the fact of recrystallization (nucleus formation and grain growth) has sensitivity limits quite different from those of the measurement of hardness, for example, which yields a summary value involving a much larger region of the testing material. This difference may also contribute greatly to the difficulty in establishing a simple interrelation between softening and recrystallization.

As Mr. Polushkin states, the recrystallization temperature range is much wider than is generally assumed. As can be seen from the author's hardness recovery curves s, t, u, Fig. 1, the extension of the recovery range of hardness also varies greatly and it would be inadvisable to use the beginning of softening as a characteristic temperature, the exact location of which is further complicated in the case of nickel by a hardness increase shortly before softening. The Tr-values falling approximately in the center of the softening range seem to be reasonably well defined. Now, if we also locate the middle of the recrystallization temperature range, I believe that Mr. Polushkin will agree

(1) that nickel certainly does not recrystallize first and then soften (Tammann) and

(2) that hardness recovery range and recrystallization range of nickel overlap but do not coincide.

The same holds for the temperature ranges in which sharpening of the Debye lines and softening takes place. In view of the difficulties involved in establishing recrystallization temperatures microscopically, recourse to X-rays should be had.

Mr. Polushkin's critical remarks are greatly appreciated, as they support very well the author's primary purpose in writing his paper, namely to indicate that the recovery of cold-worked metals on annealing actually represents one of the "most difficult problems in physical metallurgy."

Dr. Wilson's supplementary analysis of his previously studied testing material is certainly a valuable contribution. It is true that the problem of the effect of hydrogen on the physical properties of metals, and, in particular, those of nickel has not been sufficiently investigated. Dr. Wilson presents evidence supporting the assumption that such an effect does exist. According to Guichard and co-workers, 10 different electrolytic nickel preparations showed the following hardness and hydrogen contents:

Apparently there is no quantitative correlation between these 2 factors. Copper and zinc when electrodeposited contain great amounts of hydrogen but are not much harder than both metals are in the annealed state:

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H ₂ Content in Volume Per Cent	Brinell Hardness as Deposited	Annealed
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Fe 200-6000 Zn 2000 Cr 5000	140–350 40–50 500–900	69 40 140

Sieverts "loaded" remelted electrolytic iron and nickel with hydrogen (15 and 70 vol. per cent respectively) but no change in hardness took place,

The softening curve of electrolytic nickel as recently determined by Jenicek (Revue de Métallurgie, June 1936, 371) differs somewhat from that of Guichard (Fig. 4). Softening starts at slightly over 400 degrees Cent. and most of the hardness has been removed at 500 degrees Cent. Yet annealing for 20 hours at 480 degrees Cent. resulted in no microstructural changes. Slight changes were noticed in the temperature range of 550 to 650 degrees Cent.; annealing at 850 to 1100 degrees Cent. was necessary to produce coarse recrystallization. These recent experiments show, analogous to the recovery of cold work hardness and recrystallization, that the release of electrodeposition-induced hardness occurs in a lower temperature range than the elimination of the fine structure blamed for the hardening effect.

The "fine-grain size" and "hydrogen" hypotheses, however, are not the only interpretations for the unusual hardness of electrolytic nickel. Microscopic observations and annealing tests, for instance, have led McNaughton to the assumption that colloidal nickel compounds play an important rôle. Without taking any final stand in this controversy, the author feels obliged to mention that his latest experiments definitely indicate an effect of hydrogen on the recovery of nickel from cold work.

The interesting practical observations of Mr. Donachie are not in disagreement with statements of this paper. In contradistinction to observations of Tammann (on nickel) Mr. Donachie found with P-bronze that the softening temperature depends on the amount of cold work and confirms that the physical properties of the annealed state may be restored without abolishing the microscopic deformation texture. This is still contested for iron and brass, but has been definitely established for aluminum and its alloys ("Kristallerholung," "Kristallvergütung").

However, fine grain as such is no criterion as to whether recrystallization has taken place or not. As indicated by recrystallization space diagrams, in which per cent reduction is plotted against grain size and annealing temperature, the recrystallized grain becomes finer the greater the amount of plastic deformation, all other conditions being equal. With an extremely fine grain structure it becomes increasingly difficult to decide whether polyhedral grains have formed and a decision by X-rays should be made. There are many commercial applications and shaping processes in which grain size is such a vital factor that a careful establishment of a recrystallization diagram is required. The recent trend in many working processes has been to leave some "residual strength" in annealing rather than to apply a high temperature or long time anneal resulting in large grain size. Mr. Donachie's findings apparently confirm the experience of other metallurgists, namely that a better combination of strength and ductility can be obtained by cold work and annealing than by cold work alone.

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Regarding Mr. Donachie's further questions, I feel that an anneal at 1000 degrees Cent. for 1 hour should completely remove all effects of previous cold work in compact electrolytic nickel recovering from cold work several hundred degrees below this temperature. I appreciate Mr. Donachie's hint that the pretreatment, or strictly speaking, the crystal orientation of the annealed material, may affect the cold working and recovery characteristics. It is known that the Laue patterns show some residual effects of cold working even after annealing considerably above the recrystallization temperature range.

Regarding the compressed nickel powder scraped from the cathode, it can be reasonably assumed that an anneal at 1350 degrees Cent. eliminated "any residual cold work." However, it is dubious, in the light of my latest experiments, whether the origin of the sample (chips and powder) was completely obliterated. The shifting of the softening range caused by progressive cold working of nickel made from powder, and microscopic observations thereon,

will be discussed in the near future.

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THE IMPORTANCE OF BOUNDARY ATTACK IN THE ETCHING OF STEEL SPECIMENS

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By B. L. McCarthy

Abstract

This paper discusses the important part the attack of an etchant plays in determining the appearance of ferrous microstructures.

Both the boundaries of grains and constituents are considered in this discussion. The increase in solubility of the metal at the boundaries as compared with the solubility at the center of the grains or constituent particles is due to a difference in atomic activity resulting from unbalanced atoms and amorphous material.

The structure of pearlite is discussed and a reason

for the light and dark areas suggested.

Microstructures of quenched and tempered sicels are shown and the change in etching characteristics is attributed to a change in the amount of boundary material present. The darkening of the microstructure changing with the particle size of the precipitated iron carbide.

Fine pearlite associated with martensite etches out quickly because of the relative thinness of the ferrite and cementite plates which results in a large amount of con-

stituent boundary material.

The rearrangement of the relationship between crystal units produced by cold work results in an increase in atomic activity which promotes solution of the metal.

THE action of etching reagents on the surface of metallographic specimens, which brings out the structural characteristics, does not seem to be clearly established. It is the opinion of some that the light and dark areas noted are the result of a differential etching attack on the constituents, the dark areas supposedly resulting from the formation of minute terraces which do not reflect the light. Others hold that the attack on the crystalline boundaries produces minute trenches which absorb the light. Both theories have their application and indications are, that the structures developed result from a combination of both. If the attack is in the form of a light etch, and only the boundaries of the grain or constituents etch out, the second

A paper presented before the Eighteenth Annual Convention of the Society held in Cleveland, October 19 to 23, 1936. The author, B. L. McCarthy, is metallurgist, Wickwire Spencer Steel Co., Buffalo, N. Y. Manuscript received June 24, 1936.

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theory applies. If, however, the etching is carried to a point where the constituents proper are attacked the irregularities developed on the surface would produce minute terraces and the effect would be in line with the first theory. Beilby has advanced the theory that a thin coating of amorphous metal covers the specimen surface after polishing and that etching is required to remove this film. While it may be true that, due to crystal fragmentation, some amorphous material exists on the surface, its presence is not of importance in determining the etching characteristics. Employing 1 per cent nitric acid in alcohol and exposing the sample to it for only ten seconds will result in some attack on the grain boundaries. It is obvious from this that the solution of the amorphous metal if present is not important. Advanced studies of structural changes in ferrous metals using higher magnifications have necessitated the use of light etching and the most significant work today in the field of ferrous metallography, consists of a study of the boundaries of constituents as well as of grains. This applies to structures cooled at a rate that produces pearlite, also quenched and tempered specimens.

The study of crystal analysis made possible by the use of the X-ray has shown that crystals, of which all metals in the solid state are composed, consist of atoms arranged in some definite geometrical pattern and that at the boundaries of grains and constituents there exist atoms which because of their position in the space lattice are unbalanced; that is, the forces of secondary valence which hold them in position in the crystals are only partially satisfied in the case of boundary atom. This results in an increased atomic activity in these areas over that existing in the center of the grain or constituent particle. When the specimen is etched these unbalanced atoms at the boundaries are free to enter into combination with the etchant, thereby increasing the solubility of these areas.

Rosenhain and Ewen (1)¹ have shown that the crystallization of metals proceeds by the development of crystal units around a common nucleus and that the crystal units formed around a single nucleus will all be in the same plane, also that at the point where the crystal units from one nucleus, all in a given plane, meet the crystal units from another, not in the same plane, some metal is present which because of lack of space has not crystallized. This material which exists at the grain boundary retains the structure of the solute and is said to be amorphous. In these areas the full forces of secondary

¹The figures appearing in parentheses pertain to the references appended to this paper.

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valence are in effect and an increased state of atomic activity exists,

In the formation of an intermetallic compound such as iron carbide the tendency to form amorphous material at the constituent boundaries is less, yet it is conceivable that some amorphous metal exists. The size of the particle naturally determines the amount of boundary present in a given structure and would, on the whole, influence the rate of etching of the specimen. This applies to structures of pearlite, of varying degrees of fineness, as well as the particle size of cementite resulting from precipitation and coalescence.

The increased activity at the grain or constituent boundaries resulting from the atomic activity of the amorphous metal, while contributing to the rate of attack, is not in itself sufficient to account for the markings noted. Some attack on the grain or constituent surfaces at the boundaries is required to produce markings of sufficient magnitude to be visible under the microscope. The amorphous metal at the boundaries must be limited to at least two atom diameters, otherwise a unit cell would be formed and this material would no longer be amorphous.

The two constituents of steel, ferrite and cementite, when lightly etched appear white, further etching results in a differential attack, the ferrite becoming darker than the cementite. An etching too light to show this differential etching attack, is however, sufficient to bring out the constituent boundaries. This indicates definitely an increased solubility at the boundaries over that of the constituents. The appearance of a metallographic specimen under the microscope depends on the manner in which the light is reflected. If the incidence of light is perpendicular to the specimen surface, the reflection will be perpendicular and it will appear white. If, however, the incidence of light is at an angle to the surface the reflection will be at the same angle and the light will be reflected away from the eyepiece. When a metallographic specimen is etched, the surface is altered and the angle of incidence is changed depending on the nature of the attack. The solubility of the metal may result in a film or deposit on the surface which absorbs some light. However, the orientation on which the metallography of steel is based has to do more with the factors mentioned above.

In Fig. 1 is shown a structure of spheroidized cementite lightly etched, note that the cementite particle boundaries were affected by this etch but that the constituents were not. In Fig. 3 is shown a sample of low carbon steel lightly etched. Heavier etching tends to

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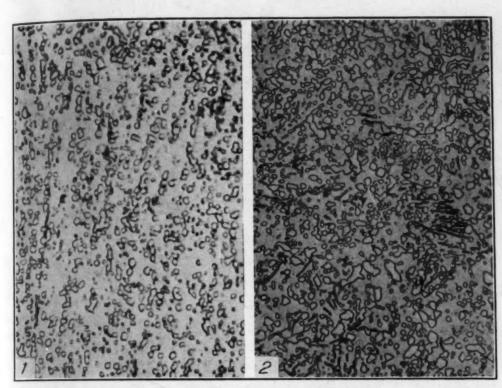


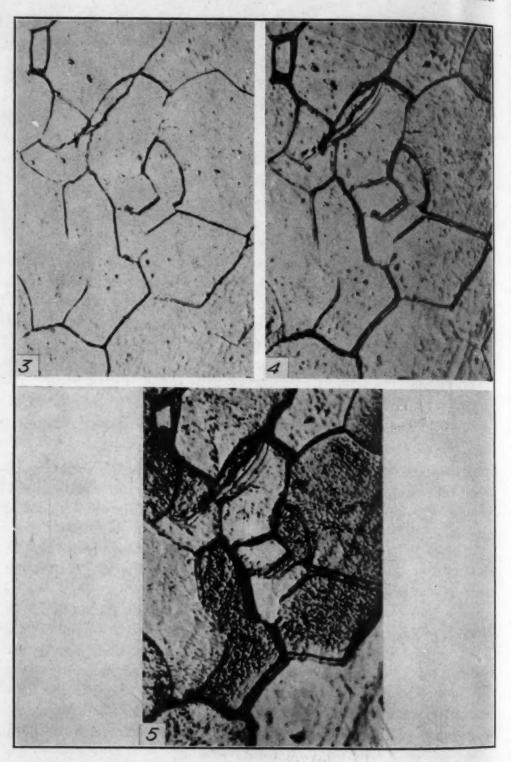
Fig. 1—Spheroidized Cementite Etched for Ten Seconds 1 Per Cent HNO₃ in Alcohol. × 1000.

Fig. 2—Spheroidized Cementite Showing Differential Attack Between Cementite and Ferrite Etched for Thirty Seconds. × 1000.

widen the dark lines at the grain boundaries. This is shown in Fig. 4. When because of the shape of a grain, a thin surface exists at the boundary it is possible by etching to dissolve away the thin edge and expose the grain underneath. This is noticeable when on deeper etching a white area appears in the grain boundary. See Figs. 4 and 5. The author's conception of the attack of the etching reagent on the boundaries is shown diagrammatically in Fig. 6.

The rate of attack on a constituent depends on its solubility in the etching reagent and the crystallographic orientation of the crystal units. Grains with crystal surfaces in a plane perpendicular to the incidence of light require more etching for visual surface alterations than do grains with a crystallographic plane at an angle to the incidence of light. This is due to the solubility of crystal fragments produced in polishing which alter the specimen surface, some light being lost due to reflection and absorption.

In Fig. 5 is shown a photomicrograph of the same area shown in Figs. 3 and 4 after heavier etching. Note the different orientation of the various grains. The shape of the grains has an influence on



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Fig. 3—Low Carbon Steel Etched for Ten Seconds. × 1000.

Fig. 4—Same as Fig. 3. Etched for Thirty Seconds. × 1000.

Fig. 5—Same as Figs. 3 and 4. After Etching for One Minute and Thirty Seconds. × 1000.

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the width of the grain boundary developed as a result of etching. Adjoining grains with surfaces perpendicular to the specimen surface seem to widen most, while it is possible to have the grain boundary almost obscure when because of the shape of the grain only a minimum amount of unbalanced grain boundary atoms is exposed to the attack of the etchant.

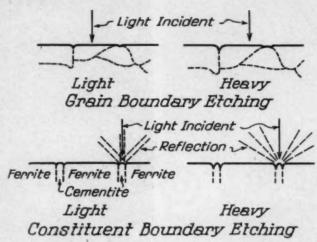


Fig. 6—Showing the Author's Conception of Etching Reagent Attack on Crystal Boundaries.

The differential etching effect between ferrite and cementite is shown in Fig. 2. Nitric acid in alcohol was employed in etching all the samples contained herein, other etching reagents such as sodium picrate, cuprous chloride, etc., are often employed for identification purposes. Some of these produce a stain effect, others are employed because of a preferential attack on certain constituents. For the general run of steel specimens a weak solution of nitric acid in alcohol seems most desirable. By its continued use, the metallographist is not so apt to become confused in his studies, and a clearer conception of structural changes resulting from heat treatment can be obtained.

In the examination of structures of pearlite the question often arises as to whether the white areas are cementite or ferrite. The etching effect which is characteristic of these structures is sometimes referred to as a differential attack on the two constituents. If this were the case and if we were to base our conclusion on a differential etching attack the conclusion could be drawn that the white areas because they are less attacked are cementite. The fact that cementite occupies only 12.8 per cent of the total pearlitic area indicates that this is not true because in all structures of pearlite, when resolved, the white areas are in the majority.

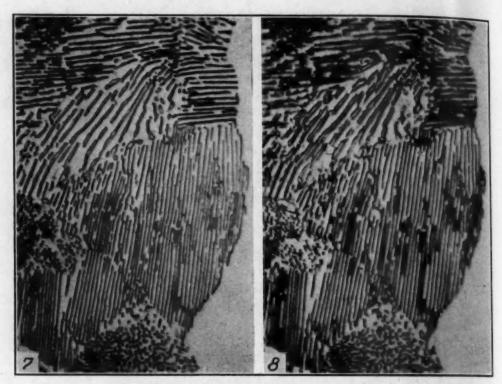


Fig. 7—Pearlite After Light Etching. Etched for Ten Seconds. × 2000. Fig. 8—Same as Fig. 7. After Medium Etch for Thirty Seconds. × 2000.

In the etching of pearlite the attack is almost entirely on constituent boundaries. If the plates of cementite are thick enough, or if the plate surfaces are in a plane other than perpendicular to the plane of the specimen surface, it is possible by light etching to bring out the constituent cementite and have it appear white with the boundaries etched. This is present in some portion of the structure shown in Fig. 7. The structure of pearlite wherein the plates are relatively thin, will, because of the constituent boundary attack, leave none of the cementite in a plane perpendicular to the incidence of light. The lines which appear dark in these structures are the result of a boundary attack on both the cementite and the ferrite plates. A study of Fig. 7 will reveal areas of this nature also. As the degree of fineness of the pearlite or the thinness of the plates increases it is possible to lose resolution even at high magnification and employing a light etch. Some of the structures shown by Vilella, Guellich and Bain (2) which they term fine pearlite, yet appear structureless, are examples of this. On prolonging the attack of the etchant a continued rounding of the edges of the ferrite and cementite at the boundary takes place and resolution may be lost even in relatively coarse pearlitic structures

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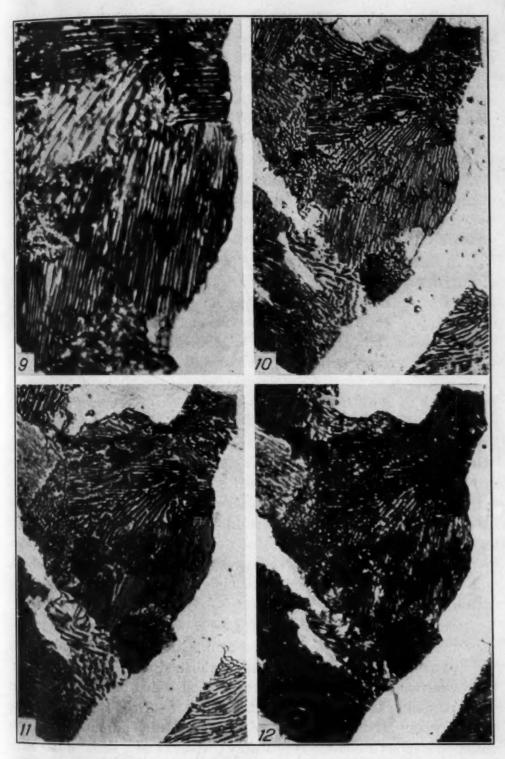


Fig. 9—Same as Figs. 7 and 8. After Heavy Etch. Etch for One Minute and Thirty Seconds. × 2000.

Fig. 10—Same as Fig. 7. Taken at × 1000.

Fig. 11—Same as Fig. 8. Taken at × 1000.

Fig. 12—Same as Fig. 9. Taken at × 1000.

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due to over-etching. This is shown in Figs. 8 and 9 which are the same structure shown in Fig. 7 after further etchings. A comparison of Figs. 7, 8 and 9 taken at × 2000 and Figs. 10, 11 and 12 taken at × 1000 will show how resolution of the structure of pearlite may be lost due to over-etching. As discussed at length by Belaiew (3) the plane in which the plate surface of the pearlite exists in relation to the plane of the specimen surface results in what appears to be variation in the thickness of the plates of ferrite and cementite. While this is true to a large extent, yet the amount of coarse pearlite present as a result of this depends on the coarseness of the pearlitic structure as a whole. As the degree of fineness of pearlite increases this will not be so apparent.

In the study of quenched and tempered specimens the effect of the attack on the constituent boundaries is of considerable importance. the etching characteristics depending on the size of the particle and the amount of constituent boundary present. Before proceeding with this discussion, however, it might be well to call attention to the importance of etching technique. We should bear in mind that we are dealing with a metal and an etching reagent which have a chemical affinity for each other and that over-etching will, by a definite attack on the constituents or by too rapid attack at the boundaries of the grains or constituents, produce a surface of no value for metallographical examination. A careful etching technique based on light etchings to bring out areas of greatest atomic activity can reveal to the metallographist valuable knowledge of the physical characteristics of the specimen. In this connection it seems advisable to consider metallography and etching as a study of atomic activity, a knowledge of which, when combined with the present theories on crystal analysis, will give a clearer conception of the changes which take place in heat treating.

The structure of martensite formerly was considered a fine dispersion of carbide particles in alpha iron. X-ray analysis has cleared this up, however, and it is now well established that it is a solid solution of carbon in both alpha and beta iron, also that two crystal phases exist, namely a cubic and tetragonal phase, (4). While the etching characteristics of martensite have not been fully accounted for the needle-like structure noted is probably the result of a differential etching attack on the two crystal phases. Steel in the martensitic condition does not etch as quickly as does the same specimen after tempering and some carbide precipitation takes place. The fact that this checks

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analysis, e in heat with X-ray studies and that as the carbide particle size grows by raising the tempering temperature, the rate of attack of the etching reagent decreases, adds strength to the theory that the etching characteristics of tempered steel depend on the amount of constituent boundary present.

When a quenched specimen is reheated the rate of attack of the etchant increases until a temperature of about 700 degrees Fahr. is reached. Further raising of the tempering temperature shows a decrease in rate of attack. This may be interpreted as indicating that complete precipitation of the carbide does not take place until the temperature of about 700 degrees Fahr. is attained. The same atomic activity which we believe influences the solubility of the boundary material has been termed surface tension and the growth of particle size which occurs as the tempering temperature is raised is said to be due to a relief of surface tension. The rigidity of the metal, at low temperatures seems to be great enough to overcome surface tension, as the temperature is raised however, these forces overcome the rigidity of the metal and there is a relief of strain by the reduction of constituent boundary material. This relief takes the form of less unbalanced surface atoms and less amorphous metal at the constituent boundaries. In the same manner the attack of the etchant is decreased in proportion to this decrease in atomic activity.

In considering the rate of etching attack on the specimen as a whole, the total effect does change with the amount of boundary present, however, there seems to be no difference in the rate of attack on the boundaries proper, the difference in etching characteristics being influenced by the particle size of the carbide and the change in the total amount of boundary. Samples which have been spheroidized will etch out at the boundaries just as quickly as samples in the tempered state, but because of the fine particle size present in the tempered samples and the tendency for the etchant to round off the corner of the boundaries, these samples will darken more. It is often the case that due to the fine particle size it is impossible to resolve certain areas. The reason for this lies in the fact that to make the boundaries visible it is necessary that there be some attack and when the particle size is extremely small its whole surface may be altered in bringing out the boundary. This produces a dark area the same as a thin plate of cementite will appear dark even after light etching.

Figs. 13, 14, 15, 16 and 17 show the effect of etching on quenched and tempered specimens. In this case the same specimen was em-

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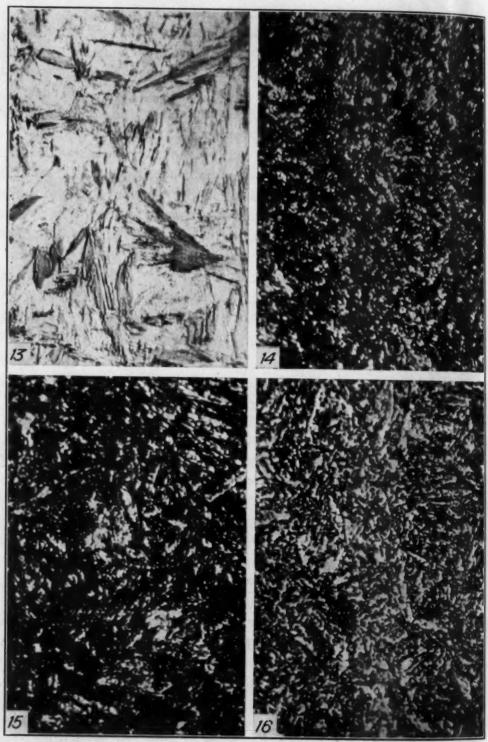


Fig. 13—0.65 per cent Carbon, 0.75 per cent Manganese Steel. Quenched in Oil From 1500 Degrees Fahr. Hardness C-62. × 2000.

Fig. 14—Same as Fig. 13. After Heating to 500 Degrees Fahr. for Thirty Minutes. Hardness C-48. × 2000.

Fig. 15—Same as Fig. 14. After Further Heating to 900 Degrees Fahr. for One Hour. Hardness C-40. × 2000.

Fig. 16—Same as Fig. 15. After Further Heating to 1150 Degrees Fahr. for Twelve Hours. Hardness C-32. × 2000.

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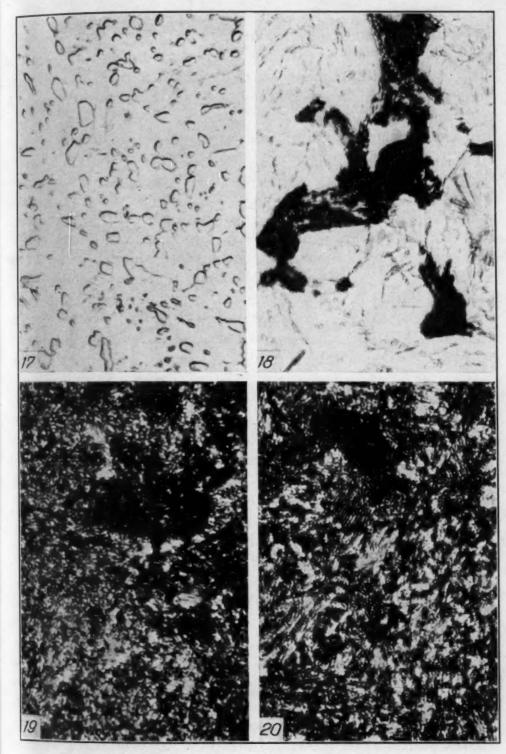


Fig. 17—Same as Fig. 16. After Further Heating to 1320 Degrees Fahr, for Twenty-Four Hours. Hardness C-12. × 2000.

Fig. 18—0.63 per cent Carbon, 0.82 per cent Manganese Steel Taper Quenched Showing Martensite and Fine Pearlite. × 2000.

Fig. 19—After Reheating Fig. 18 to 500 Degrees Fahr, for Thirty Minutes. × 2000.

Fig. 20—After Heating Fig. 19 to 920 Degrees Fahr, for Thirty Minutes. × 2000.

ployed throughout, care being taken to avoid decarburization or carbon absorption on heating. An examination of these photomicropgraphs will show an increase in particle size as the tempering temperature is raised, also a marked change in etching characteristics. The treatment employed on the specimen shown in Fig. 17 corresponds to a spheroidizing treatment.

The nodular structure found associated with martensite which has been shown to be fine pearlite has etching characteristics different from those of the average pearlitic structure or of tempered steels. In this case the extreme fineness of the cementite and ferrite plates results in the formation of a large amount of boundary material and they etch out quickly. That this structure in spite of the amount of boundaries present is much more stable than the quenched and drawn structure can be seen by an examination of the photomicrographs shown in Figs. 18, 19, 20, 21 and 22. When associated with martensite in the quenched condition the fineness of the plates is at a maximum. Heating to 500 degrees Fahr. however, affords some relief of surface tension and we find that the structure after this treatment does not etch out as dark. That it is still darker than the tempered martensite is shown in Fig. 18. That this structure is more stable than the tempered martensite is shown by the photomicrographs in Figs. 18 and 19. This tendency toward greater stability is undoubtedly due to the inability of the plates to coalesce readily into rounded particles. That this finally takes place is shown in Fig. 21. This photomicrograph shows definitely the influence of particle size on the etching characteristics.

The difference in the behavior of the nodular structure on tempering is important in considering the fatigue life of tempered steels. Inasmuch as fatigue life is largely dependent on the uniform distribution of stress it is easy to believe that where a marked difference in structural characteristics exists there would be lack of uniform stress distribution. Your attention is called to the area of pearlite in Fig. 20. In this connection it seems important that serious thought be given to the effect of retained austenite on fatigue life. While the author has not done sufficient work to justify a conclusion, indications are that at a tempering temperature of 900 degrees Fahr. some of the retained austenite may transform directly to fine pearlite. This being the case it is logical to assume that the characteristics of a tempered specimen could be appreciably aftered by the amount of austenite retained.

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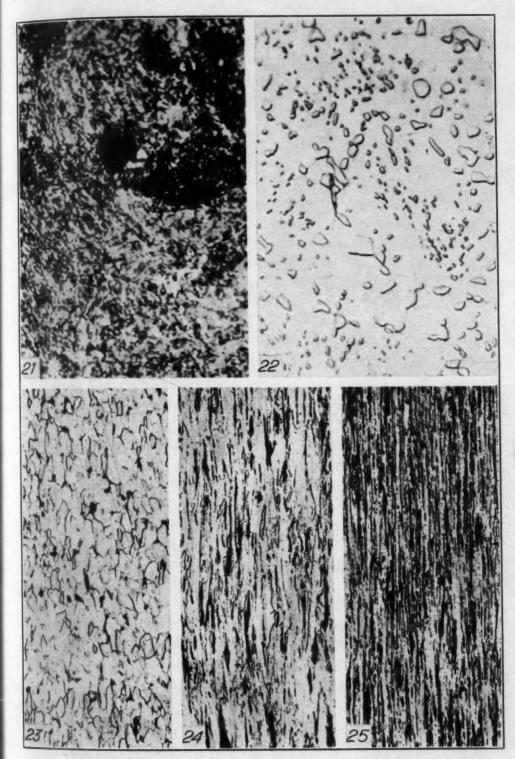


Fig. 21—After Heating Fig. 20 to 1150 Degrees Fahr. for Two Hours. × 2000. Fig. 22—After Heating Martensite-Fine Pearlite to 1310 Degrees Fahr. for Twenty-Four Hours. × 2000. Fig. 23—Cold Drawn Wire Reduced 25 Per Cent. Etched for One Minute. Hardness B-82. Tensile Strength 82,000 Pounds Per Square Inch. × 200. Fig. 24—Same as Fig. 23. After 72 Per Cent Reduction Etched for One Minute. Hardness B-92. Tensile Strength 104,500 Pounds Per Square Inch. × 200. Fig. 25—Same as Fig. 23. After 92 Per Cent Reduction Etched for One Minute. Hardness B-103. Tensile Strength 126,000 Pounds Per Square Inch. × 200.

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The relation of particle size to hardness has been accounted for by the slip interference theory (5), that the etching characteristics are definitely in line with the amount of boundary material present and the hardness is shown by the hardness values given. The length of time at temperature when hardened steel is being tempered is important. Basing our opinions on the particle size developed and the hardness values, indications are that as the tempering temperature is raised, the time at temperature should be increased to establish equilibrium for a given temperature. The reason for this change in required time-at-heat in tempering is probably due to a diminishing of force as the surface tension is relieved and the tendency toward the reduction of boundary material by coalescence is lessened.

In studying cold drawn specimens the rate of attack of the etchant varies with the amount of deformation. This is shown in Figs. 22, 23 and 24. Samples, which because of excessive cold work have been subject to considerable slip along crystallographic planes. will etch out more quickly. The reason for this can again be accounted for by the degree of atomic activity. The phenomenon of cold working hardness has been covered by Jeffries and Archer and others (5). It has been shown that deformation results in innumerable slips along crystallographic planes and while continued cold work results in resistance to slip, yet it is obvious that any deformation must be accompanied by some slip. When due to slips along crystallographic planes the relation of the atoms of one series of cubes to the atoms of another is altered, the balanced state of the atoms adjoining the slip is likewise altered. In the same manner as in the case of boundary atoms we have an accelerated attack or increased solubility resulting from this increase in atomic activity. Another factor that influences the etching of cold drawn structures is the increase in grain boundary material in a given area. Due to deformation the grains are elongated and while there is no increase in the total number of grains there is an increase in the number present in any given area whether the specimen is viewed transversely or longitudinally.

That amorphous metal is produced as a result of cold work and accounts for the hardening of cold-worked metals has been advanced by Beilby. While the author favors the slip interference theory of Jeffries and Archer (5) as a more suitable explanation of this phenomenon, in either case, an increase in atomic activity would result, and would account for the accelerated attack of the etchant.

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same factors noted above. As the carbon content is raised the rate of solubility is increased due to an increase in constituent boundary material.

The increase in solubility resulting from cold work is likewise traceable to the change in atomic activity noted above.

These statements, of course, apply to plain carbon steels only. The addition of alloying elements which are soluble in the ferrite may so alter the solubility as to overcome the influence of boundary material. In this connection the author refers particularly to the stainless steels whose solubility is influenced by the amount of chromium in solution in the ferrite.

Conclusions

The structural charactertistics, as observed under the microscope, which result from etching are a result of both an increased rate of attack of the etchant on the constituent and grain boundaries as well as on the constituents. The boundaries because of an increased atomic activity etch out first.

While the amorphous metal present at the boundaries contributes to the increased rate of attack of the etchant, the amount of this material present is not sufficient to account for the markings noted. There must be some attack on the surface of the grains or constituents to make the markings visible.

The dark lines noted in pearlitic structures are the result of a boundary attack between the two constituents. The thickness of the cementite plate determines whether or not it is visible under the microscope.

The slower rate at which martensite will etch out as compared with tempered structures is due to the absence of constituent boundaries. Indications are that precipitation of iron carbide from martensitic structures is not complete until a temperature of about 700 degrees Fahr. is reached.

The etching characteristics of tempered specimens depends on the particle size of the carbide, the darkening of the sample diminishing as the particle size increases.

Indications are that the nodular structure recently shown to be fine pearlite is more stable than the martensitic areas surrounding it.

The fatigue properties of quenched and tempered steels may be influenced by the amount of austenite retained on quenching.

The increased solubility of cold drawn specimens is due to the existence of a greater atomic activity resulting from the change in the relations between the atoms of the cubes adjoining the planes along which slip has taken place and to an increase in boundary material present in a given area.

The solubility of steel when considered generally is influenced by the amount of constituent boundaries present.

The author wishes to express his appreciation to the Wickwire Spencer Steel Co., in whose laboratory the data and photomicrographs employed herein were developed.

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- (2) Vilella, Guellich and Bain, "On Naming the Aggregate Constituents of Steel" Metal Progress, Vol. XXVIII, August, 1935.
- (3) N. T. Belaiew, "The Inner Structure of the Pearlitic Grain", Journal, Iron and Steel Institute, 1922, No. 1, Vol. CV.
- (4) Kotaro Honda and Zensi Nishiyama, "On the Nature of the Tetragonal and Cubic Martensite". Transactions, American Society for Steel Treating, Vol. XX, November, 1932.
- (5) Zay Jeffries and R. S. Archer, "Slip Interference Theory of the Hardening of Metals", Chemical and Metallurgical Engineering, June 15, 1921, Vol. 24, p. 1057.

DISCUSSION

Written Discussion: By Morris Cohen, Instructor, Division of Physical Metallurgy, Massachusetts Institute of Technology, Cambridge, Mass.

In spite of the fact that the basis of the science of metallography depends largely upon the appearance of metallic structures as revealed by the application of an etching reagent to a polished surface, we generally pay little attention to the actual mechanism of etching. Many of us who examine steels etched with nital (nitric acid in alcohol) as a part of daily routine are too prone to accept the microstructures as a matter of course without considering what makes these structures visible. The present paper demonstrates how much more information can be obtained from the metallographic examination of even ordinary steels when the mechanism of the nital attack is known.

With respect to the etching of pearlitic structures, the author has shown the existence of chemical attack at the ferrite-cementite interface. However, there is another, and perhaps more important effect of nital etching which was not mentioned. In any heterogeneous structure consisting of both hard and soft constituents, there is a tendency for the soft regions to be worn down preferentially during the polishing operation, leaving the harder particles standing out in relief.

Simultaneously, however, the valleys thus formed are filled in with debris and become covered over by metallic flow to the extent that the polished sur-

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However, which was hard and worn down icles stand-

with debris lished surface may appear quite smooth even at high magnification. This is very much the case with polished pearlite where the soft ferrite valleys in between the hard cementite ridges become filled and smoothed over with the result that the lamellar structure is not visible in the unetched condition. The nital reagent when applied for even a short length of time cleans out these valleys. This action can be demonstrated in the following way: Fig. A of this discussion shows an edge view of four sheets of low carbon steel which were pinched together and polished. The lines of demarcation between the four

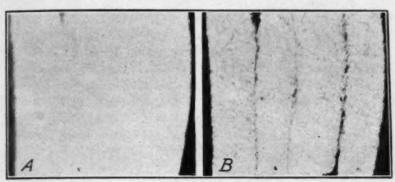


Fig. A—Four Sheets of Low Carbon Steel Pressed Together and Polished. Unetched. Lines of Demarcation Between Sheets Invisible Due to Surface Flow.

Fig. B—Same as Fig. A Lightly Etched with 3 Per Cent Nital. Lines of Demarcation Between Sheets are Clearly Visible Due to the Solution of Flowed Metal.

sheets are not visible. After light etching, however, each interface becomes clearly visible (Fig. B) due to the solution of the debris and flowed metal which has collected between the sheets during polishing. The ferrite which flows is either severely distorted and fragmented or is amorphous, which would account for the high rate of chemical attack. The system of hills and valleys thus disclosed in pearlite acts as a diffraction grating and produces an iridescent play of colors when viewed at the proper angle with the naked eye. This gives the structure its characteristic pearly luster.

Further evidence of this action of nital is shown in Fig. 17 on page 1069 of the author's paper. Here the line of demarcation between the cementite particles and the ferrite matrix is most sharply defined along the upper and left-hand edges of each particle. This is due to slightly oblique illumination casting shadows of the hard cementite particles standing in relief. In the unetched condition, most of these particles would have been covered over with ferrite which flows readily in the polishing operation, and the action of the nital is principally to dissolve this flowed metal. If the hard particles were in the same plane as the ferrite matrix, and the nital simply attacked the ferrite-cementite interface, as suggested by the author, the boundaries of the spheroids in Fig. 17 would be more uniform in spite of the oblique illumination.

In Figs. 4 and 5 on page 1062, some of the grain boundaries contain white streaks which the author believes are due to grains underneath showing up through the deeply attacked boundaries of the surface grains. It is more probable, however, that these white streaks are merely due to reflected light coming from the bottom of the troughs formed in the deeply etched boundaries.

I have noticed this "double boundary" in specimens of pure copper deeply etched with nitric acid, and found that the position of any given white streak within a grain boundary could be shifted laterally at will by varying the angle of incident illumination. This would indicate that the light streak in a deeply etched boundary has no special structural significance. However, the author's interpretation could be tested in a simple but critical fashion by polishing away successive layers and re-etching each time to see if the white area in a particular boundary becomes wider as more of the postulated grain beneath is revealed.

Written Discussion: By R. Schempp, metallurgist, Halcomb Steel Co., Syracuse, N. Y.

Mr. McCarthy bases his etching theory upon the fact that "The size of the particles determines the amount of boundary present in a given structure and would, on the whole, influence the rate of etching in the specimen." This may theoretically be true in any alloy or metal that does not possess allotropism, but in any material that possesses an allotropic transformation, or in any metastable system, this theory is not applicable since other factors tend to influence and determine the final etchability of the steel. This is clearly illustrated by means of the corrosion test which really is a quantitative etching test carried to completion.

The corrosion results of numerous investigators show that the corrosion loss per unit area of a quenched steel increases with tempering up to approximately 100 degrees Cent. (212 degrees Fahr.) and, then, with further tempering, gradually decreases until it reaches a minimum at about 150 degrees Cent. (300 degrees Fahr.). Additional tempering again increases the corrosion loss until a maximum is reached in the osmonditic state, which exists at a temperature range of 300 to 400 degrees Cent. (570 to 750 degrees Fahr.), depending upon the temperature time. Tempering above this temperature range progressively decreases the corrosion loss until it reaches a minimum in the spheroidized state. Thus, these results readily indicate that the particle size does not ultimately determine the etching characteristics, or the corrosion loss, of any material in a metastable condition but is overshadowed by the physical properties of the transforming aggregate.

Regarding Mr. McCarthy's statement on page 1070 that "at a tempering of 900 degrees Fahr, some of the retained austenite may transform directly to fine pearlite," it seems quite contrary to the general theory and conception of pearlite which postulates that pearlite only results when austenite is cooled through the thermal critical range at a rate less than that of the critical quenching speed which depresses the Ar' to approximately 300 degrees Cent. (570 degrees Fahr.)—to form martensite. Furthermore, the state of division of the pearlite (cementite lamellae) is determined by the rate of cooling through the critical as oil quenching produces the finest state of division (troostite), and furnace cooling—the coarsest cementite lamellae in pearlite. It also postulates that when austenite is retained in a drastically quenched steel (martensitic), it decomposes directly upon tempering into a supersaturated solution of carbon in alpha iron with submicroscopic precipitation of the carbon of iron carbide in the crystallographic planes and axes of the alpha iron. Further tempering completes precipitation and causes agglomeration of

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the precipitated particles to a microscopical size which appears as nodular troostite upon etching. Since this nodular troostite is formed by the agglomeration and coagulation of the precipitated carbide particles, they form along the planes and axes of the crystallographic lattice in a manner typical of fine pearlite but, actually, are fine individual, separated spheroidal carbide particles and not cementite lamellae which denotes the structural characteristics of troostite which is obtained by oil quenching.

Although the writer's views are somewhat different from Mr. McCarthy's regarding this subject, he wishes to congratulate him upon the excellent manner in which he presented this very debatable question and he trusts that the author will continue his experiments in order to clear up the moot points of this important subject.

Written Discussion: By E. R. Carr, Jr., research metallurgist, Crucible Steel Company of America, Pittsburgh.

In this paper, Mr. McCarthy has done a noteworthy piece of work on some rather fundamental ideas. A continuation of topics of this sort would,

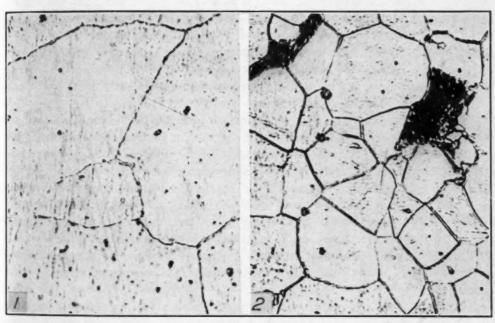


Fig. 1—Armco Ingot Iron Etched in 5 Per Cent Nital for 10 Seconds. X 1000. Fig. 2—Carbon Steel Having 0.12 Per Cent Carbon Etched in 5 Per Cent Nital for 10 Seconds. X 1000.

it is believed, lead to a better understanding of the process and thereby of the technique of etching. It is, however, desired that the author clear up a few points which are not entirely self-explanatory.

It is understood from the paper that the author is speaking of all constituent boundaries when he says that they etch most rapidly. This agrees with the facts brought out in the paper except that no mention is made of the relative rates of attack of cementite particle boundaries and ferrite grain boundaries, and the two seem to be used more or less indiscriminately.

Many times when specimens of spheroidized tool steel are badly over-

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etched, the grain boundaries of the ferrite present as a matrix begin to be shown up. Such a thing would seem to indicate that either Fig. 2 of the author's paper shows only more complete cementite boundary attack than Fig. 1 and not true ferrite attack; or that the constituent ferrite is attacked before the ferrite grain boundaries. However, from Figs. 3, 4, and 5 of the paper it appears that the latter supposition is rather impossible as Fig. 3 shows ferrite grain boundaries but no ferrite attack while Fig. 5 shows very decided ferrite constituent attack.

The present writer would like to suggest that possibly such a difference in susceptibility to the etchant of the ferritic grain boundaries and cementitic particle boundaries may account for the difference in etching time between various hypoeutectoid steels. The majority of workers in this field have no doubt noticed at one time or another the great difference in the time that is required to etch a piece of Armco ingot iron and, say, a piece of 0.10 per cent carbon steel. Such a case is illustrated in Figs. 1 and 2 of this discussion respectively. In Fig. 1, a piece of Armco iron is shown which has been etched for ten seconds in 5 per cent HNO₂ in ethyl alcohol. This shows the ferritic grain boundaries of this material not quite completely etched. A piece of 0.12 per cent carbon steel is shown in Fig. 2 also etched for ten seconds in the same concentration of nitric acid in alcohol as the Armco iron. Here the ferritic grain boundaries are already becoming over-etched due, it is believed, to minute carbide particles along these ferritic grain boundaries which etch more readily than the ferrite boundary constituent.

In regard to the author's conception of pearlite, the writer is heartily in accord with the idea that the white areas shown on nital etching are ferrite for the most part. As the author states, where the cementite plates are of sufficient thickness the cementite will show white with the boundaries etched. The writer has always looked at the pearlitic structure from the point of view that it consisted of grains of ferrite in which plates of cementite are embedded; these plates being precipitated from the austenitic grain during its change to the ferritic phase. It is felt that such an expression of the structure as opposed to the conception of pearlite as alternate plates of cementite and plates of ferrite would lead to a clearer idea of the mechanism of the formation of the eutectoid.

Mr. McCarthy has performed an unusual piece of work on a subject which has been rather neglected. A closer understanding of the mechanism of such etching action and the possible standardization of various etching solutions would, no doubt, lead to a clearer interpretation of microstructures by metallographers in general.

Oral Discussion

J. E. Wilson: I have listened to Mr. McCarthy with a great deal of interest. He has made a number of pertinent observations regarding the technique of etching steel. Without wishing to appear hypercritical, I do feel that attention should be called to the fact that the photomicrographs reproduced in Figs. 8, 13, 18, 19, 20 and 21 are slightly off-focus. This condition makes it

¹Metallurgist, Bausch & Lomb Optical Co., Rochester, N. Y.

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difficult to verify the findings regarding effect of etching time on apparent resolution without repeating the tests.

Was a standard etching time used for the cold drawn steel wire specimens? Due to the well known fact that cold working markedly increases the corrosion rate of steel,² the etching time should be decreased, particularly for specimens severely worked such as those represented by Figs. 24 and 25.

I should also like to question Mr. McCarthy concerning his exact procedure of polishing and the reagents used in preparation of the 1 per cent nital. I ask this because it has been my experience that the polishing method of Urban and Schneidewind³ gives less surface flow with steel specimens than the conventional loose powder methods. With respect to the etching reagent, the use of fuming HNO₂ with absolute ethyl alcohol gives a much sharper grain boundary attack and a lesser tendency toward general attack than ordinary concentrated HNO₂ in 96 per cent alcohol.

Author's Reply

Mr. Cohen's remarks relative to the wearing down of the softer areas when pearlitic structures are being polished are well taken and are, I believe, the generally accepted views. The degree of variation in the wearing down of softer areas depends of course on the polishing technique. As I visualize proper polishing, it should consist of an abrasive or cutting action with only enough pressure being applied to the specimen to afford contact with the polishing cloth. This would practically eliminate relief polishing effects. It has always been my feeling that the amount of wearing down of softer areas and the filling in with debris has been greatly magnified in the explanation of it. Certain it is that this effect is being greatly reduced by the improvements in polishing. It has been argued by some that the collection of debris or an amorphous coating obscures the grain boundaries and that etching, by clearing away the debris and dissolving the amorphous covering, makes the grain boundaries visible. This is an exaggerated application of this theory for in the absence of any debris or amorphous metal some etching attack at the boundaries would be needed to make them apparent. I am sure that a more careful polishing of the three samples of strip shown by Mr. Cohen would make the lines of demarcation visible after polishing. I do not feel that polishing a number of soft pieces of strip steel is quite the same as polishing a specimen containing areas of pearlite. In the latter case, the hard constituent of cementite would tend to prevent the flow of the ferrite and would promote the cutting or abrasive action.

Regarding the so-termed pearly appearance of the structure of pearlite, this could result from a reflection of light from the sides of the valleys produced by etching. That the development of this pearly appearance is in line with the amount of etching lends strength to the belief that this is the case.

I have attempted to follow out Mr. Cohen's suggestion regarding the test to determine the reason for white lines in the grain boundaries, by repolishing. While I was unable, after numerous trials, to demonstrate the presence of a

Tammann and Moritz, Annalen der Physik, Vol. 16, 1933, p. 567.

³S. F. Urban and R. Schneidewind, Metal Progress, American Society for Steel Treating, Aug. 1932, p. 39.

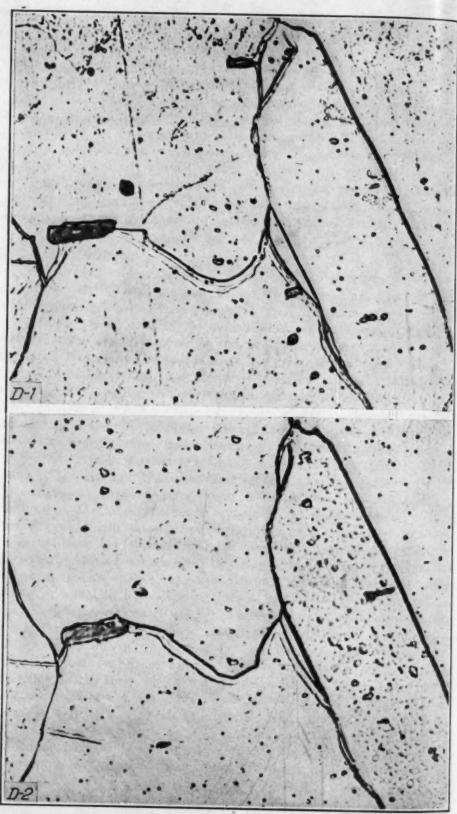


Fig. D-1—0.08 Per Cent Carbon Steel Etched 3 Times for 10 Seconds with 5 Per Cent Nital. Washed After Each Etching. × 1000.

Fig. D-2—Same as Above After Repolishing for 5 Minutes Using Wet Magnesia. Etched Same as Above. × 1000.

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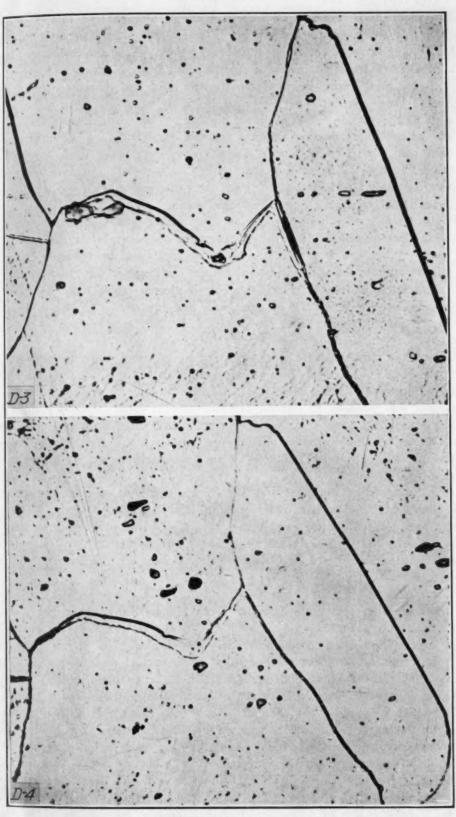


Fig. D-3—Same as Fig. D-2. After Further Repolishing for 5 Minutes Using Magnesia and Same Etching Procedure. × 1000.

Fig. D-4—Same as Fig. D-2 After Further Repolishing and Etching as Fig. D-3. × 1000.

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grain lying beneath the surface with a grain surface parallel to the specimen surface, this work, photomicrographs of which are shown in D-1, D-2, and D-3, D-4, has brought out some interesting points. Note that what appears to be a number of small grains is in reality small, thin fragments of grains cut from the original grains in preparing the specimen and that these small grain fragments have beneath them other grains with a surface in a plane almost parallel to the plane of the specimen surface. The white lines discussed are present around these grain fragments and are the result of a sufficient amount of light being reflected back into the microscope to make them appear light in color. It appears from this that while the postulated grain beneath may account for the white markings in some cases, the majority of the white markings can be accounted for in the following manner:

Because of the polygonal shape of the grains, grain boundaries lie in a varied number of planes. The nearer the plane of the grain surface approaches the plane of the specimen surface, the more light will be reflected back into the microscope and the wider will be the white markings in the grain boundaries. The dark lines on each side of the white markings result from an attack of the etchant on the boundary of one grain and the corner of the other grain where the plane changed from the plane of the specimen surface to the plane of the grain surface which lies beneath the adjoining grain.

These photomicrographs were prepared by repeated polishing and etching. The polishing consisted of five minutes on broadcloth using wet magnesium oxide. The speed of the wheel was 350 revolutions per minute. Nitric acid in alcohol was used as an etchant and all samples were etched for three tensecond intervals followed by washing and drying between each etch. Note how the nonmetallic inclusion was reduced in thickness and finally removed. It was necessary to select an area of this nature to retain its identity after repolishing.

This work does not bear out Mr. Cohen's contention that the reflected light comes from the bottom of the troughs formed on deep etching. The only bottom that I can visualize must come from a grain beneath for otherwise the trough would be V-shaped and no light would be reflected. I would like, however, to express my appreciation to Mr. Cohen for this very helpful and constructive discussion.

Mr. Schempp's findings regarding the increase in corrosion loss on increasing the tempering temperatures range from 300 to 750 degrees Fahr. checks with the author's findings on etching. Also the decrease in corrosion loss when higher tempering temperatures are employed. No attempt has been made to comment on the solubility of steels quenched and tempered at tempering temperatures below 300 degrees Fahr. The changes which take place on tempering the metastable structure of martensite at these low temperatures could readily account for the changes in corrosion loss found by Mr. Schempp.

I do not agree with Mr. Schempp regarding the generality of the theory and conception of pearlite which postulates that pearlite only results when austenite is cooled through the thermal critical range at a rate less than that of the critical quench speed. While I am in accord with the theory and work of Bain and his associates there are many who still hold to the older theories on the transformation of austenite. Further, the true mechanism of the trans-

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on increashr. checks cosion loss has been d at temtake place inperatures Schempp. the theory ults when than that and work er theories the transformation of retained austenite is not clearly understood. The transformation of austenite is accompanied by a volume increase and it is quite possible, in the author's opinion, that the retained austenite would transform direct to pearlite in the case of small sections of wire being tempered in a continuous operation. In this operation the wire of small diameter runs directly from an oil quenching bath held at 150 degrees Fahr. to a lead bath held at about 1000 degrees Fahr. Any delay in the transformation of austenite because of lack of space for volume increase could result in its occurring at a temperature sufficiently high to produce pearlite.

The question of the relative rate of attack between the boundaries of ferrite grains and cementite particles, brought out by Mr. Carr, is indeed worthy of consideration. The author has noted that the grain boundaries of ferrite in spheroidized structures seem to etch out slower than the ferrite grain boundaries of steel which have not been held at the spheroidizing temperature. There does seem to be a difference in the rate of attack on cementite particle boundaries as compared with ferrite grain boundaries. It would be very interesting to have this difference established because it undoubtedly has an influence on the etching characteristics of pearlite and spheroidized structures.

Mr. Carr's comments on the difference between the rate of attack on the ferrite grain boundaries of Armco iron as compared with ordinary low carbon steels are interesting. This is undoubtedly influenced by the purity of the metal. I have noted a marked difference in the rate of attack between the ferrite boundaries of rimmed steel found at the center and in the rimmed area and have always attributed this to the difference in amount of impurities.

Mr. Carr's suggested manner of expressing the structure of pearlite is indeed worthy of thought, for, is not the cementite plate nothing more than a shape assumed by the carbide at the time of precipitation from austenite and is it not also a precipitated constituent in a ferrite matrix?

I wish to take exception to Mr. Wilson's statement that Figs. 8 and 13 are off-focus. If Mr. Wilson has had much experience in trying to focus steels such as those shown in Figs. 18, 19, 20 and 21 when lightly etched, at 2000 diameters, he should appreciate the difficulties encountered. All these samples under normal conditions of etching would be much darker. The light etching produces a number of small rounded particles with wide valleys which makes the selection of a proper focus difficult. As to the accuracy of the focus, I naturally thought they were well focused or they would not have been used.

I do not agree with Mr. Wilson that the etching time of steel decreases with an increase in corrosion rate. As far as I can determine, this is contrary to fact and contrary to the findings of Mr. Schempp as shown in his discussion of this paper. Specimens used in this paper were polished by the wet powder method. The method of polishing employed by Urban and Schneidewind has an application when nonmetallic inclusions and graphite particles are being studied but the author prefers the wet powder method for the examination of microstructures.

As stated in the paper, I am not in favor of varying the etchant for each individual study. When one is employing metallography in an effort to solve metallurgical problems, it seems much easier to employ a solution of concentrated nitric acid in ethyl alcohol.

AN X-RAY STUDY OF PREFERRED ORIENTATIONS IN PURE COLD-ROLLED IRON-NICKEL ALLOYS

By D. McLachlan, Jr., and Wheeler P. Davey

Abstract

Specimens of very pure Fe-Ni alloys up to 21.66 per cent nickel have been prepared, with a high degree of homogeneity. These were rolled into ribbons about 10 x 0.5 millimeter in cross section and were then annealed to 850-900 degrees Cent. They were then measured and cold-rolled, samples being cut off at about every 15 per cent reduction in thickness starting at about 30 per cent. The rolling treatment was varied with respect to (1) per cent total reduction, (2) per cent reduction per pass, (3) temperature of the rolls, and (4) direction of rolling. The specimens were studied by X-ray diffraction methods (1) to determine the effect upon the preferred orientation of (a) nickel content, (b) per cent reduction, (c) large versus small reductions per pass, (d) temperature, and (2) to find how deeply these effects extend below the surface. The experimental results have been expressed by the customary stereographic pole-figures, and, starting with a very simple picture of the mechanism of rolling of body-centered cubic crystals, theoretical equations have been derived which are capable of predicting the experimental results.

THE work of Wever and others on the preferred orientation of crystals in rolled iron sheets is too well known to need comment or even a summary. Insofar as the writers have been able to find, however, nothing has been reported in the literature on cold-rolled sheets of iron-nickel alloys. Such alloys are especially interesting as representing one extreme type of solid solution, namely that of a continuous range of solid solutions embracing two crystal

Contribution from the Division of Physical Chemistry and Chemical Physics, School of Chemistry and Physics, The Pennsylvania State College, State College, Pa.

A paper presented before the Eighteenth Annual Convention of the Society held in Cleveland, October 19 to 23, 1936. The material here presented is from a thesis presented by D. McLachlan, junior author, in partial fulfillment of the requirements for the Ph.D. degree at Pennsylvania State College; W. P. Davey is Research Professor of Physics and Chemistry, The Pennsylvania State College. Manuscript received May 29, 1936.

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forms, yet showing no thermal or X-ray evidence of a separate phase of intermetallic compound. Such a series of alloys would be doubly interesting if they were so free from impurities that all experimental results could be safely interpreted in terms of the iron and nickel atoms alone. The writers believe that they have made Fe-Ni alloys of this degree of purity and of an extraordinary degree of homogeneity. It is the purpose of this article to give the results of X-ray diffraction experiments on the preferred orientations in the cold-rolled sheets of such alloys, and to endeavor to give a tenable theory which serves to correlate the experimental facts.

PREPARATION OF ALLOYS

Pure iron was made by the following method: A jet of a solution of ferric nitrate in distilled water was caused to swirl in air around and in contact with a similar jet of synthetic ammonia dissolved in distilled water. The resulting precipitate was washed free of electrolyte, dried to oxide, reduced to metallic powder in a rotary silica furnace in a stream of purified hydrogen, formed into a bar 1/4 x 1/4 x 4 inches in size under a pressure of 30 tons per square inch, sintered in hydrogen, hot-swaged to wires 1/8 inch in diameter, and annealed in pure hydrogen above 850 degrees Cent. The total impurities in a representative rod made by this method were found by spectroscopic methods to be less than 40 parts per million. Fe-Ni alloys were made in exactly the same way except that the solution of ferric nitrate was replaced by a solution of a mixture of iron and nickel salts. It is obvious that every step in the process tends to promote greater and greater homogeneity of composition of the alloy. This is consistent with the results, as yet unpublished, of a microscopical examination by S. D. Smith on specimens made by this method.

The ½-inch wires were rolled into ribbons about 0.4 inch (1 centimeter) wide and 20 mils (0.5 millimeter) thick. These ribbons were heated in pure hydrogen for six hours at temperatures above 850 degrees Cent. in order to remove such impurities as might diffuse to the surface and combine with the hydrogen. The ribbons were then cooled slowly by turning off the heat from the furnace. X-ray diffraction patterns were made of these ribbons in order to be sure that the annealing had completely destroyed the effects of mechanical working in the rolls, and to be sure that the cooling in

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the furnace had been slow enough to prevent strains. Samples of each of these randomly oriented strain-free specimens were taken for chemical analysis for nickel content by the dimethylglyoxime method. Still other samples were cold-rolled according to various techniques, thus providing specimens for X-ray examination. More than 70 specimens were examined, giving a total of over 450 diffraction patterns. The rolls were 2¾ inches (7 centimeters) in diameter and the linear rolling speed was 6½ inches per minute (16.2 centimeters per minute). This slow speed was used in order to prevent local heating of the specimen due to rolling.

EXPERIMENTAL APPARATUS AND TECHNIQUE

Successive passes through the rolls for a given specimen were made in the same direction and with the same face against the upper roll. Thicknesses were measured before and after each pass, using micrometer calipers. For each specimen the number of passes and, except where otherwise specifically noted, the per cent reduction for each pass were recorded. "Reflection" X-ray diffraction patterns were taken for each specimen and "transmission" patterns were taken of the thin specimens. For each setting the two angles, (1) between the X-ray beam and the plane of the specimen, and (2) between the X-ray beam projected on the plane of the specimen, and the "transverse" direction of rolling, were predetermined so as to be of greatest service in making a stereographic pole-figure. These angles were selected with the aid of a special automatic stereographic pole-figure machine.

X-rays were produced by a water-cooled Coolidge molybdenum target tube with ZrO₂ filter, operating at 30 KV, and 25 MA. Fluorazure intensifying screens were used with Eastman duplitized ultra-speed safety X-ray films. The specimen to film distance was approximately 4.5 centimeters. Provision was made for oscillating the specimen in its own plane in order to obtain a fair "sampling" of the specimen. All orientation studies were made on the original negatives, positive prints being made only for special purposes. When positive prints were made at all, they were made either with the aid of a rotating sector with a 100-watt lamp, or with a 7-watt lamp placed directly above the center of the arcs of the diffraction pattern at a distance of from 2 to 6 inches from the glass of the printing frame.

¹D. McLachlan, Jr., Rev. Sci. Inst., Vol. 7, 301, 1936; Zeit. f. Kryst, 1936.

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The intensities of the various portions of the arcs in a given diffraction ring on a negative are usually taken as a criterion of the relative number of crystallographic planes having corresponding orientations in the metal sheet. This, of course, is correct only (1) when absorption of X-rays in the specimen is taken into account, and (2) when it is remembered that zero blackness on the negative represents only that number of planes so oriented is too small to give enough X-ray intensity to affect the photographic emulsion. These two sources of error may be provided against sufficiently if we compare intensities only within individual arcs, and if we express all intensities for a given arc in terms of the intensity of the blackest portion of that arc. This assumes that no part of the arc is completely opaque. The blackness of the various portions of a given arc may be translated sufficiently well into intensity of the diffracted X-ray beam by comparing the negative with calibrated test strips of the same type of X-ray photographic film, using a properly adjusted intensity of diffused light. It was found convenient to mount these calibrated test strips in the form of a fan, something like the "feelers" of a machinist's thickness gage.

Pole-Figure Technique

"Monochromatic pinhole" diffraction patterns yield both direct and indirect information as to the orientation of crystals. Having once picked out the type of plane-family whose orientations are to be plotted, (say the {110} form), it is possible to obtain direct information from the positions and angular lengths of the arcs in the diffraction ring corresponding to that family of planes. This direct information may be transferred at once to the stereographic polefigure by means of the McLachlan projection machine¹ mentioned above. The spots on the diffraction arcs of still other types of plane families, (say the {100} and/or {211}, may be made to yield indirect information as to possible additional positions of {1 1 0} poles. This is because the plane-families which give rise to these other rings belong to still other crystals in the specimen which must be so oriented as to give the correct Bragg angle for diffraction for these plane-families. The ability to translate the data from the {100} and/or {2 1 1} types of planes into data for the {1 1 0} form depends upon a knowledge of the angular relationships between the planes in question, and on the ability to think of the diffraction data in terms of the crystallites in the rolled specimen sheet.

The angular relationships between plane-families in a single cubic crystal are shown in inverse perspective in Fig. 1a, and in stereographic projection in Fig. 1b. Fig. 1c gives the angular scale for Fig. 1b. It remains to relate the diffraction data of certain plane-families, (say the {100} and/or the {211} types), to the ori-

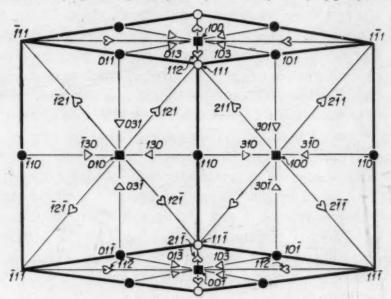


Fig. 1a—Inverse Projection of Cubic Crystal Showing Intercepts of Plane-Normals with Cube-Faces. The Observer is Looking into the *Interior* of the Cube.

entations of the crystallites in the specimen and to record these orientations in terms of stereographic projections of the {1 1 0} poles. The procedure may be illustrated as follows: Consider a specimen of rolled sheet, a photographic film, and an X-ray beam, as they exist together in a diffraction experiment. (See Fig. 2.) Let the film be perpendicular to the incident beam, as usual. Let the rolling direction in the specimen be horizontal and perpendicular to the incident beam, and let the transverse direction make an angle a with the incident X-ray beam. Imagine a vertical plane to be passed through the incident beam and imagine a second plane passed through the incident and diffracted beams. These two planes cut the photographic film in two straight lines which make an angle which we shall call Δ . A plane passed through the normal to the diffracting plane and through the rolling direction will cut these same two planes in two lines which form an angle which we shall call \(\tau \). Since \(\tau \) is the projection of Δ on the plane passing through the normal to the diffracting plane, we have

$$\sin (90^{\circ} - \tau) = \sin \Delta \cos \theta$$

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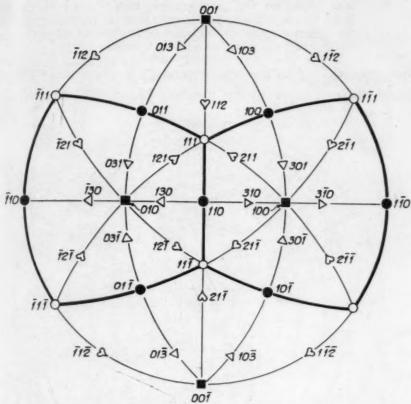


Fig. 1b-Stereographic Projection of Plane-Families of a Cubic Crystal.

where θ is the Bragg angle of diffraction. We have already imagined a plane drawn through the rolling direction and the normal to the diffracting plane. Now imagine another plane drawn through the rolling direction and the normal to the specimen. Call the angle between these two planes, Ψ . Then

$$\cos (\Psi - \alpha) = \frac{\cos \Delta \cos \theta}{\cos (90^{\circ} - \tau)}$$

This enables us to find both τ and Ψ in terms of a, θ , and Δ . In other words, we can determine how the crystallite is oriented with respect to the surface of the sheet by a calculation involving only the experimentally known values of:

- a = the angle between the incident X-ray beam and the rolled sheet. Its sign is positive if the beam hits the upper surface of the specimen; negative if it hits the lower surface.
- θ = the Bragg angle for the family of planes used.

The angle on the photographic film formed by drawing two lines through the central spot; one, a vertical reference line; the other, passing through the photographic record of the beam diffracted by the crystallite in question.

The changes required in the above equations when the rolling direction is not perpendicular to the incident beam will be obvious.

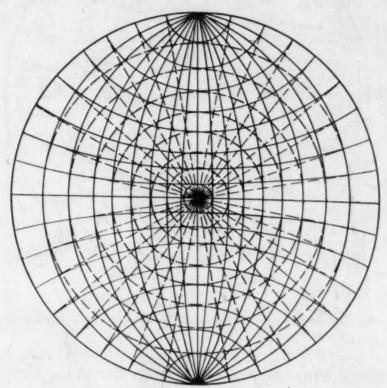


Fig. 1c-Angular Scales for (b).

If the specimen is mounted to comply with the limitations stated, the above equations apply to any one of the crystallites which help to produce some portion of a given diffraction arc. If the angle Δ is measured from the vertical reference line to the nearest end of the arc we shall call it Δ_n ; if measured to the far end of the arc we shall call it Δ_t ; if measured to the middle of the arc we shall call it Δ_m . These give three values of τ , namely τ_n , τ_t , and τ_m . It is not necessary to calculate separate values of Ψ from τ_n and τ_t for they would turn out to be too close together for separate plotting. It is sufficient to calculate a single value of Ψ from the value of τ_m . The values of τ_n , τ_t , and Ψ from each arc in a given diffraction ring can then be translated into the corresponding values for a $\{1\ 1\ 0\}$ pole by the aid of Figs. 1b and 1c. This is easiest done by moving, in Fig. 1b, the pole for the diffracting plane to the position τ_m , Ψ . This

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causes the poles for the $\{1\ 1\ 0\}$ type of planes to move to new positions τ_m' , Ψ' ; τ_m'' , Ψ'' ; etc. Then the pole of the diffracting plane is moved to τ_n and τ_t , and the poles of $\{1\ 1\ 0\}$ are necessarily moved by the same angular amount. These latter values can then be trans-

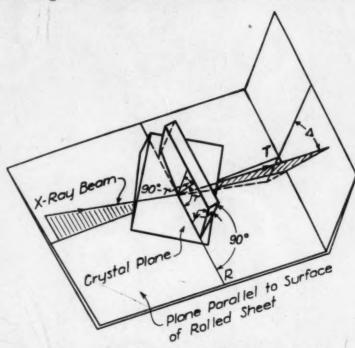


Fig. 2—Perspective View of Angles Δ , Ψ , and τ .

ferred directly to the McLachlan projection machine and plotted on the pole-figure. The use of the projection machine would not be necessary at this point if Fig. 1b were on exactly the same scale as the projection made by the machine.

ORIENTATION DATA AND THEIR INTERPRETATION

Each specimen was assigned a code symbol for note-book reference. The code was such that the members of a series of specimens, designed to bring out the effect of some one variable, had some portion of the symbol in common. Such a series could, therefore, be designated by that common portion of the symbols of its members. Each diffraction pattern was marked not only with the code symbol of the specimen but also with additional symbols which expressed the details of the X-ray experiments.

Series A included a total of 53 specimens which varied in nickel content. The composition, original thickness, number of passes through the rolls, the final thickness, and the total per cent reduction

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Table I
Rolling Schedules of Specimens Having Various Nickel Contents

Code Designation	Nickel Content Per Cent	Original Thickness (mm.)	Number of Passes Through Rolls	Final Thickness (mm.)	Per Cent Total Reduction
Designation A0 A1 A2 A3 A4 2A0 2A1 2A2 2A3 2A4 2A5 3A0 3A2 3A3 3A4 3A5 5A0 5A1 5A2 5A3 5A4 6A6 6A1 6A2 6A3 6A4 6A6 7A0 7A1 7A2 7A8 7A4 7A5 8A0 8A1 8A2 8A3 8A4 8A5 9A0	Per Cent 0 0 0 0 2.10 2.10 2.10 2.10 2.10 2.10	(mm.) 0.47 0.50 0.47 0.48 0.42 0.425 0.425 0.425 0.425 0.425 0.425 0.50 0.50 0.50 0.50 0.50 0.50 0.42 0.42 0.42 0.42 0.42 0.42 0.42 0.4	Through Rolls 0 9 12 18 19 0 1 4 7 11 15 0 3 6 10 16 12 0 3 5 8 11 13 0 2 4 7 12 18 0 6 11 17 25 31	(mm.) 0.47 0.39 0.275 0.17 0.04 0.425 0.40 0.19 0.14 0.11 0.045 0.50 0.33 0.25 0.15 0.11 0.055 0.42 0.32 0.25 0.20 0.14 0.06 0.47 0.36 0.28 0.22 0.16 0.13 0.56 0.49 0.31 0.22 0.16 0.13 0.55 0.49 0.31 0.22 0.16 0.10 0.47	Reduction 0 22.0 41.5 64.6 90.5 0 5.9 76.5 89.5 0 34.0 50.0 70.0 78.0 90.0 0 23.8 41.5 52.4 66.7 85.6 0 23.4 40.5 53.1 66.0 72.3 0 12.5 44.6 60.6 71.5 89.0 0 45.5 65.6 74.6 82.0 89.0
9A ⁰ 9A ¹ 9A ² 9A ⁸ 9A ⁶ 10A ⁰ 10A ¹ 10A ² 10A ⁸ 10A ⁴	15.5 15.5 15.5 15.5 15.5 21.66 21.66 21.66 21.66 21.66	0.47 0.47 0.47 0.47 0.47 0.50 0.50 0.50 0.50 0.50	0 8 12 17 22 34 0 2 7 14 21 31	0.47 0.28 0.20 0.15 0.11 0.05 0.30 0.20 0.15 0.11	0 41.5 57.5 68.2 76.6 89.4 0 40.0 60.0 70.0 78.0 88.0

for each specimen are listed in Table I. Since this series was rolled at an early stage in the work, no record was made of the reduction per pass. This, however, turns out in the light of later work not to have been a serious omission. The diffraction patterns of this series, when translated into the customary pole-figures, show no systematic change in preferred orientations with increase in nickel

Per Cent Total Reduction 0 22.0 41.5 64.6 690.5 0 55.9 76.5 89.5 0 34.0 70.0 78.0 90.0 23.8 41.5 52.4 40.5 66.6 60.6 72.3 12.5 44.6 671.5 89.0 0 41.5 57.5 68.2 76.6 89.0 0 41.5 57.5 68.2 76.6 89.0 0 40.0 60.0 70.0 88.0

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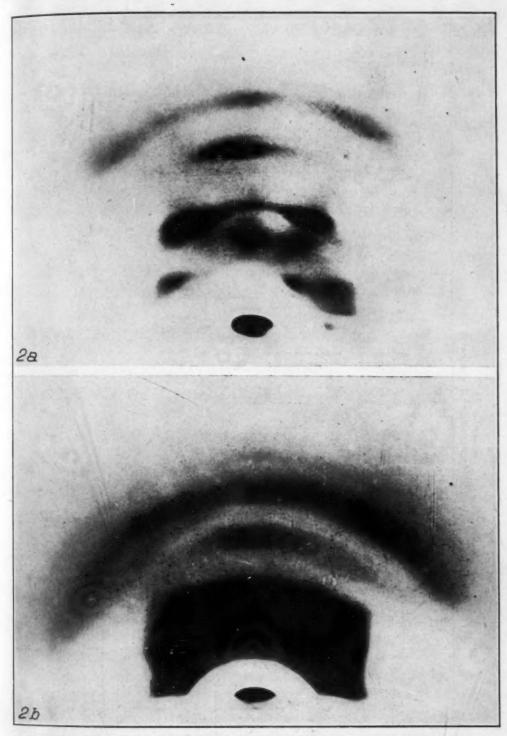


Fig. 2a—Monochromatic Pinhole Pattern of Sample 2 Taken at 10° Angle. Fig. 2b—Monochromatic Pinhole Pattern of Sample Q Taken at 10° Angle.

content. Those patterns taken by the "transmission" technique merely confirmed the well-known fact that the greater the total per

	Total %	65.3 87.3			880.64 6.05 6.05 6.05 6.05 6.05 6.05 6.05 6.05
Sample	% Reduction	65.3 37.5			0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0
	Thickness	.205			7 2 2 CC
	Total %	0.000.000.000.000	77.4		Sample 333
mple Z	% Reduction Per Pass	28.50	26.9		
Š	Thickness	25222	.113		
	Total % Reduction	0 62.3 75.4 883.6 55.3	888.5		*
Sample	% Reduction Per Pass	0.00 13.3 10.0 10.0	6.3		
02	Thickness	162111100	.0075	1	
	Total %	00000000000000000000000000000000000000	66.7 69.0 72.6	82.0	
ample Y	% Reduction Per Pass	15.6	13.3	16.6	
S	Thickness	24.4. 23.3. 27.5.	1335	.075	
	Total % Reduction	0.24.20 25.07.184	55.3.88.89.9 59.3.88.89.9	73.0 73.0 73.0 73.0 73.0 73.0 73.0 73.0	2.4.
Sample X	% Reduction Per Pass	44.200	10.4 10.4 12.8	8.2.01. 2.0.4.4. 3.0.7.6.4.4.4.4.4.4.4.4.4.4.4.4.4.4.4.4.4.4	130
	Thickness	24.4.4.8.8.8.8.8.8.8.8.8.8.8.8.8.8.8.8.8	.24 .215 .195	125	290.
	Total %	0128.8.4.9.4.6.1.3.3.4.6.1.3	7.4 9.0 11.5 29.5	28.45.65 28.47.46.85	882.0 87.0 87.0 87.0 87.0 87.0 97.0
Sample	% Reduction Per Pass	0.0000000000000000000000000000000000000	2.7.7. 1.8.7.7.4.0	0.40.0.4.	
	Thickness	.61 .595 .590 .590 .585	86.8.8.8.8.8.8.8.8.8.8.8.8.8.8.8.8.8.8.	444 www.	011789555
	Total %				4.08.08.08.08.08.08.08.08.08.08.08.08.08.
Sample	% Reduction Per Pass	0.00.01	1.882.0	9.1.0	vwow.4844n:1000 vwr.40468000wr.
		2222288	ກູ່ ທ່ານ ທ່ານ ໝານ 4 ພ ບ	2448 248 248 248 248	2
	No. of Passes in Rolls	0-0040	00000	154431	30 22 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2

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cent reduction, the shorter the arcs on the diffraction rings, (i.e., the higher the degree of preferment of orientation). Other patterns taken by the "reflection" technique seemed to show no correlation of any sort.

This led to series B which was intended to determine the influence of per cent reduction per pass. Samples of ribbon all of the same nickel content, (2.10 per cent nickel), and all of the same thermal history were cold-rolled to as nearly as possible the same total per cent reduction, but differing from sample to sample in the number of passes through the rolls, and therefore in the per cent reduction per pass. Table II gives the rolling history of each sample in series B. Diffraction patterns of the "transmission" type showed similar arcs for all of these samples but patterns of the "reflection" type showed a higher degree of orientation the greater the number of passes through the rolls. Taking the extreme cases, (see Fig. 2a and 2b), sample 2 showed a high degree of orientation on the "reflection" patterns, while sample Q showed a low degree of orien-This effect was more and more marked the smaller the grazing angle of the specimen. All this was consistent with the explanation that the effect of per cent reduction per pass lay chiefly in the surface of the rolled specimen. To check this conclusion, the surfaces of samples 2 and Q were etched 1.3 mils (0.033 millimeter) and 1.4 mils (0.035 millimeter) respectively, using a special nitric acid etch.* After etching, the two specimens, (which before had given such widely different orientation results), gave identical "reflection" type diffraction patterns.

In order to make sure that the results of Series B were not due to some characteristic of the nickel content (2.10 per cent nickel), two additional samples, called 5 and 7 were run with 5.33 per cent and 17.18 per cent nickel content respectively. The rolling technique was, as nearly as possible, identical with that used for sample 2. The X-ray investigation before and after etching gave results identical with those found for 2.

In all the above we had assumed that our rolling speed, (approximately 6 inches per minute), was so slow that the rate of input of mechanical work was not sufficient to cause appreciable

^{*}The surfaces of our pure iron and iron-nickel alloys can be etched very smoothly by immersing the specimen in highly concentrated HNO₃ to which a few drops of distilled water have been added. (The exact amount of water is best determined by trial.) The surface of the specimen must be touched with an aluminum rod to start the reaction. When the desired amount has been etched off, the reaction is quenched by transferring the specimen to very highly concentrated acid, thus putting the surface into the passive state. The specimen is then washed with water.

Table III
Rolling Schedule of Specimens RC and BC

Number of Passes Through Rolls	Thickness	Sample RC Per Cent Reduction Per Pass	Total Per Cent Reduction	Thickness	Sample BC Per Cent Reduction Per Pass	Total Per Cent Reduction
0 1 2 3	0.50 0.32 0.28 0.26	36.0 12.5 7.1	0 36.0 44.0 48.0	0.495 0.235 0.135 0.100	0 52.5 44.7 23.0	0 52.5 73.6 79.8
5 6	0.24 0.23 0.22 0.205	7.7 4.2 4.4 6.8	52.0 54.0 56.0 59.0	Q.070	30.0	85.8
8 9	0.195 0.18	4.9 7.7	61.0 64.0			
10 11 12	0.17 0.16 0.145	5.6 5.9 9.4	66.0 68.0 71.0			
13 14	0.135 0.125	6.9 7.4	73.0 75.0			
15 16 17	0.115 0.105 0.10	8.0 6.7 5.0	77.0 79.0 80.0			
18 19 20 21	0.09 0.085 0.08 0.075	10.0 5.6 5.9 12.5	82.0 83.0 84.0 86.0			

Table IV

Composition and Physical Treatment of Etched Specimens Used in Final Test on
Per Cent Total Reduction vs. Degree of Orientation Below Surface Skin

		Rolling Treatment					
Sample	Per Cent Nickel	Details		Per Cent Reduction			
A ¹ A ² A ³ A ⁴	Pure Fe Pure Fe Pure Fe Pure Fe	No care taken to regulate per cent reduction per pass.	9 12 18 19	22.0 41.5 64.6 90.5	Measured 13 10 3		
21	2.10 }	Very small per cent reduction per pass.	{ 43 28	87.0 74.2	4 6		
S	2.10	Large per cent reduction per pass.	9	89.5	3		
T	2.10	Medium per cent reduction per pass.	29	87.0	4		
Q	2.10	Largest possible per cent reduction per pass.	3	87.3	4		
5 51	5.33	Very small per cent reduction per pass.	{ 50 28	85.5 66.0	5 8		
5A ² 5A ³ 5A ⁴ 5A ⁵	5.33 5.33 5.33 5.33	No care taken to regulate per cent reduction per pass.	{ 2 4 7 15	41.5 52.4 66.7 85.6	13 11 7 5		
7 71	17.18 } 17.18 }	Very small per cent reduction per pass.	§ 57 28	85.4 66.1	5 8		
10A ¹ 10A ² 10A ⁸ 10A ⁴ 10A ⁵	21.66 21.66 21.66 21.66 21.66	No care taken to regulate per cent reduction per pass.	$ \left\{ \begin{array}{l} 2 \\ 7 \\ 14 \\ 21 \\ 31 \end{array} \right. $	40.0 60.0 70.0 78.0 88.0	14 10 6 5 3		

local heating of the specimens. We therefore rolled two more samples, (RC and BC), duplicating as nearly as possible the rolling

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treatment of samples 2 and Q except that during the entire rolling process dry ice was kept in contact with the exit side of the rolls. The rolling schedules for specimens RC and BC are shown in Table III. Between passes the specimen was placed on dry ice. We believe that during the entire rolling, the rolls and the specimen were never hotter than 0 degrees Cent. The X-ray diffraction results for specimens RC and BC were identical with those for specimens 2 and Q. We were therefore certain that our specimens rolled at room temperature were really "cold-rolled."

In view of the above we felt justified in concluding that the orientation of the crystallites in the center of a cold-rolled sheet of

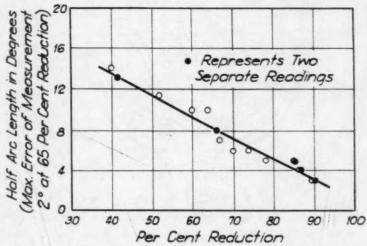


Fig. 4—Straight Line Relationship Between Per Cent Total Reduction and Degree of Orientation Below the Surface Skin. (Specimens Represent a Variety of Nickel Contents and of Physical Treatment.)

pure Fe-Ni alloy is independent of (1) the nickel content, at least up to 21.55 per cent nickel; (2) the per cent reduction per pass; (3) the temperature of the rolls, (at least up to room temperature). Within these same limits, the orientation of the crystallites in the center of a cold-rolled sheet of pure Fe-Ni alloy depends primarily upon the per cent total reduction. If these two conclusions are correct, we should be able to etch off the surfaces of the samples of both Series A and Series B, and should be able to show that the degree of preferment of orientation is a function only of per cent total reduction in thickness. This was done as follows: Specimens were taken from Series A and B which represented the greatest extremes in nickel content and in physical treatment. These were etched as described above and then X-ray diffraction patterns were taken using

as nearly as possible the same angular position with respect to the X-ray beam. These specimens are listed in Table IV. The angular half-lengths of the arcs of the diffraction rings of the {1 1 0} type of

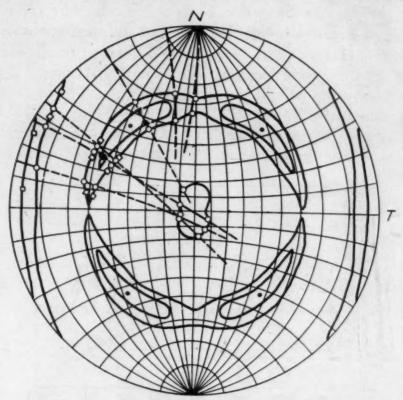


Fig. 5-Typical Pole Figures for {1 1 0} Planes for 90 Per Cent Total Reduction.

● Points of Maximum Pole Density.

▲ Points of Half-Maximum Pole Density.

o Limits of Measurable Pole Density (Obtained by Direct

Limits of Measurable Pole Density (Obtained by Indirect

Information).
O Points Between Which a Measurable Pole-Density Was Found. o Points Between Which Pole Density is Immeasurably Small.

plane families were plotted against per cent total reduction. Fig. 4 shows that, within the precision to which the arc lengths can be measured, i.e., to within a limit of 2 degrees at 65 per cent total reduction), a straight line is obtained.

We may therefore conclude definitely that, (except for a thin surface skin whose thickness is of the order of 1 mil, (0.02 millimeter) or less, the degree of preferred orientation of the crystals of our cold-rolled specimens of pure, homogeneous Fe-Ni alloys depends only upon the per cent total reduction.

A typical pole figure for {1 1 0} planes for 90 per cent total re-

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duction is shown in Fig. 5. It shows the mean orientation of the crystallites to be that in which a (001) plane-family is parallel to the surface of the rolled sheet and in which one of the face-diagonals of the unit-cube lies parallel to the direction of rolling. As is well known this result may be found repeatedly in the literature for cold-rolled iron of commercial purity. We are now able to say that this preferred orientation is characteristic not only of pure iron itself but also of homogeneous pure iron-nickel alloys, at least up to 21 per cent nickel, even when cold-rolling is rigorously defined.

If the degree of orientation is measured as the reciprocal of the true angular lengths of the arcs in a given diffraction ring, then,

within experimental error,

L-C = A-BR

where L is the length of the arc

C is the finite size of the X-ray slit system projected in the direction of the diffracted X-ray beam. L-C is the true angular length of the arc.

R is the per cent total reduction

A and B are constants.

It should be noted that the graph of Fig. 4 is made to stop at 90 per cent total reduction. This is because the course of the graph beyond that point is not known. The graph as drawn in Fig. 4, when extrapolated to 100 per cent reduction, does not approach the zero of ordinates to within half the projected width of the pinhole of the slit system.

THEORETICAL DISCUSSION

Apparently no amount of rolling will produce complete orientation of the crystallites. It would seem, then, that increased per cent total reduction merely increases the probability that a given crystallite will assume the optimum orientation. This gives a clue to the type of equation which must be used to account for the shapes of our pole-figures for various per cents of total reduction in thickness. Let us assume, now, a sphere of reference with a given crystallite at its center. We have already defined two angles, τ and Ψ , (see Fig. 2), which are sufficient to give the location of the pole of a crystal plane on the sphere of reference. Then the number of poles that will probably lie in some specified area on the surface of the sphere of reference can be represented by an expression which involves both τ and Ψ . In mathematical symbols, the expression may be written

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$$\Phi(\tau), f(\Psi).$$

Since the same number of poles can also be represented by the product of "number of poles per unit area" (= D) times the area, we have

$$D = \frac{\Phi(\tau), f(\Psi)}{\text{area}}$$

It is this quantity, D, which we must learn to calculate by finding what to substitute for Φ (τ) and f (Ψ), since a pole-figure represents, in effect, a graphical representation of the value of D for each element of area in the upper hemisphere of the sphere of reference.

The above statements are perhaps a little too simple for work with most metals, since they apply rigorously only to crystal planes having only one plane-family to the plane form. In the case where there are several plane-families per form, as in the cubic system, the final equation and the final calculations are complicated by having to account for contributions made toward the final pole-density by all the poles from each plane family of the given form.

We shall now define certain quantities in preparation for finding appropriate expressions for Φ (τ) and f (Ψ). Let us imagine a reference sphere drawn about that portion of the cold-rolled sheet which is traversed by the X-ray beam. Let the radius of the sphere be large in comparison with the dimensions of the trace of the incident X-ray beam on the specimen, so that no appreciable error is introduced by considering every part of the specimen traversed by the beam to lie at the center of the sphere. In the crystallites traversed by the X-ray beam let us pick for discussion the planes belonging to the families of some one plane-form. For instance, if we choose the {110} form, we would have the (110), (101), (011), (110), (101), and (011) plane-families. Each of these plane-families is composed of a large number of parallel planes. Let each of these individual planes be imagined to be marked off into tiny unit areas. These unit areas may be imagined for convenience to be several Angstroms across. Now let a normal be erected at the center of each of these tiny unit-areas. Such a normal is a "unit pole." If there are N unit-areas in the path of the incident X-ray beam there will be N unit-poles cutting the surface of the reference sphere. The number of poles which cut the surface of the sphere at a given point is therefore a measure of the amount of diffracting material having that parby the prodhe area, we

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ticular orientation. The average pole-density on the reference sphere is the average number of poles per square centimeter of spherical surface, i.e.,

 $\overline{D} = \frac{N}{4 \pi r^2}$

where r is the radius of the sphere in centimeters. N may be considered to be the product of three quantities: v, which is the number of plane-families in the plane-form under consideration (i.e., 6 for the $\{1\ 1\ 0\}$ form); n, which is the number of crystallites in the volume traversed by the X-ray beam; and \overline{A} which is the average area, per crystallite, of the surface belonging to any one plane-family in the plane-form chosen for discussion. We may therefore write

$$\overline{D} = \frac{v n \overline{A}}{4\pi r^2}$$

The actual pole-density, D, of a randomly oriented metal would be constant over the entire surface of the reference sphere. The process of rolling a metal should not alter the average pole-density \overline{D} , but should cause the absolute value of D to vary from point to point on the reference sphere. Our problem is, then, to solve for the values of D at any point Ψ , τ on the reference sphere.

As has been stated before, the *mean* orientation produced by the cold-rolling of pure iron and pure homogeneous iron-nickel alloys requires the crystallites to have their $(0\,0\,1)$ planes parallel to the surface of the rolled sheet and to have one of the face-diagonals of the unit-cube parallel to the direction of rolling. We shall represent the orientation of the crystallite by the orientation of the pole of its $(0\,0\,1)$ family of planes. This requires the two coordinates Ψ and τ . In the general case it would be necessary to specify three coordinates to express completely the orientation of a plane. Two of these would have to do with the orientation of the pole itself and the third with the orientation of the plane about the pole. The use of the third coordinate is obviated in the present discussion by making three simple assumptions as to the mechanism of rolling of a body-centered cubic material. These assumptions are:

- (1) A crystallite can only rotate about the face-diagonals of its unit cube;
 - (2) At high per cent total reductions, rotation takes place

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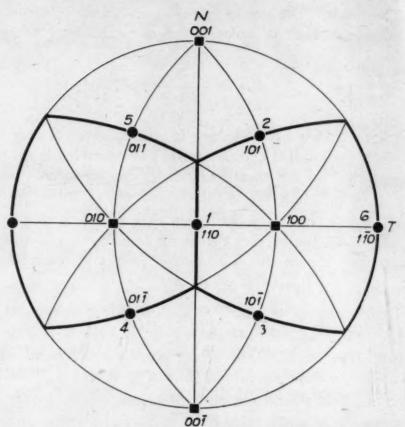


Fig. 6—Stereographic Projection of 1 1 0 and 1 0 0 Plane-Forms of a Crystallite in a Sheet of Cold-Rolled Body-Centered Cubic Metal.

about the two face-diagonals which lie closest to the rolling plane;

(3) At low per cent total reductions, the combination of rotations must be such as to so orient the crystallite that, by obeying assumption (2) the crystallite can finally put itself in the mean orientation for high per cent total reduction,—i.e., (001) plane-family parallel to the rolling plane and face-diagonal of the unit-cube parallel to the rolling direction.

These three assumptions have the practical advantage of making it possible to express the amount by which the orientation of a given crystallite of heavily cold-rolled body-centered cubic material differs from the mean orientation by using only two quantities, Ψ and τ . They are made plausible by the fact that they seem to be consistent with the structure of the crystal and with the forces involved in rolling.² They are finally justified by the fact that they lead to equations which give calculated pole-figures which duplicate those found by experiment.

²J. S. Coswell, Proceedings, South Wales Institute of England, Vol. 42, 1936, p. 201.

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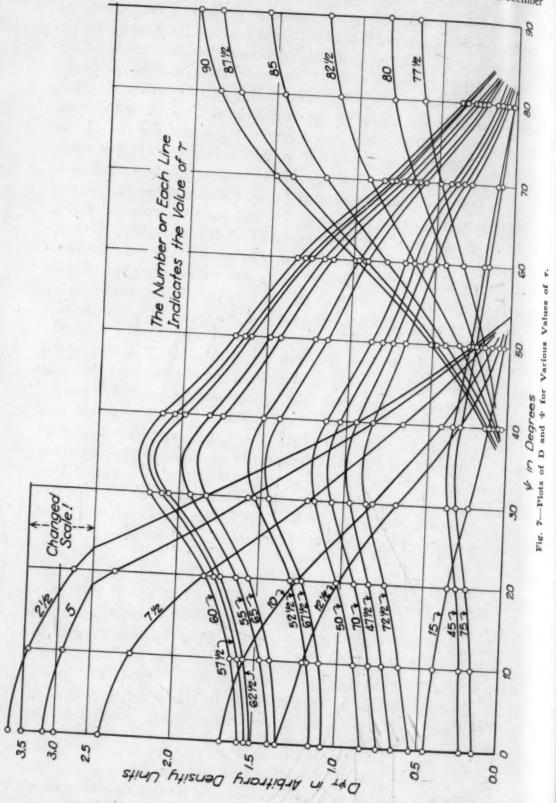
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Fig. 6 represents the stereographic projection of the various important planes of a crystallite in the mean position for heavily worked cold-rolled iron. The pole which represents the orientation of the crystallite itself is shown at N. That this is so is brought out by the following statements: (1) the pole for the (001) family of planes lies on the boundary circle of the projection and points in the direction of the normal to the rolled sheet; (2) the pole for the (110) family of planes lies at the center of the projection so that it represents a face-diagonal of the unit-cube pointing in the direction of rolling. We shall take the normal to the rolled sheet as our zero of angular measure for Ψ , (see Fig. 2), and we shall take the rolling direction as our zero for the orientation of our crystallite. The direction of Ψ will be considered positive when it represents clockwise rotation to an observer looking along the rolling direction. This gives a clockwise rotation about the (110) pole in the stereographic projection of Fig. 6. Fig. 2 shows that the quantity τ represents a rotation of a radius vector about that face-diagonal of a unit-cube which would coincide with the transverse direction of rolling if the (001) plane of the crystallite happened to lie parallel to the surface of the rolled sheet. We shall take as our zero of angular measurement of \(\tau \) that face-diagonal of the crystallite which acts as the axis of rotation for Ψ . The direction of τ will be considered positive when it represents a clockwise rotation to an observer looking along the face-diagonal in the direction toward the source of X-rays. This gives a motion in the direction towards the bottom of the paper in the stereographic projection of Fig. 6. Using these conventions of signs, the coordinates of the (001) pole of the crystallite in Fig. 6 are $\Psi = 0$: $\tau = -90^{\circ}$.

It will be remembered that it was found convenient to plot our stereographic pole-figure of Fig. 5 on the basis of the {110} form because of the large amount of direct information which we could obtain from the {110} diffraction arcs. In order that we may be able to compare our calculated pole-figures with Fig. 5 it will be necessary to make our calculations on the basis of the {110} poles. Fig. 6 shows that, when the crystallite has its characteristic pole at N, there will be six {110} poles in our stereographic projection. In Fig. 6 these are labeled 1 to 6 inclusive.*

^{*}At first sight it would look as though there were seven $\{1\ 1\ 0\}$ poles shown in Fig. 6. It is evident, however, that the $(\bar{1}\ 1\ 0)$ pole is only the other end of the $(\bar{1}\ \bar{1}\ 0)$ pole.



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If, now, the characteristic (001) pole of the crystallite is moved an angular distance Ψ' from N, followed by an angular distance τ' then each of the six $\{110\}$ poles will be moved.

Pole No. 1 will move from $\Psi = 0$; $\tau = 0$ to $\Psi = \Psi_1$; $\tau = \tau_1$. A study of Fig. 6, or of a crystal model, shows that in the case of pole No. 1, $\Psi_1 = \Psi'$ and $\tau_1 = \tau'$. We may therefore write, for the new orientation, that

Pole No. 1 has coordinates Ψ_1 , τ_1 , = Ψ' , τ' .

Similarly

Pole No. 2 has coordinates Ψ_2 , $\tau_2 = \Psi' + 35^\circ$, $\tau' + 60^\circ$

Pole No. 3 has coordinates Ψ_3 , $\tau_3 = \Psi' + 145^{\circ}$, $\tau' + 60^{\circ}$

Pole No. 4 has coordinates Ψ_4 , $\tau_4 = \Psi' - 145^{\circ}$, $\tau' + 60^{\circ}$

Pole No. 5 has coordinates Ψ_5 , $\tau_5 = \Psi' - 35^\circ$, $\tau' + 60^\circ$

Pole No. 6 has coordinates Ψ_6 , $\tau_6 = \Psi' + 90^\circ$, $\tau' + 90^\circ$

This may be expressed in general language by saying that pole k has coordinates Ψ_k , $\tau_k = \Psi' + c_k$, $\tau + C_k = \Psi' + (\Psi_k - \Psi')$, $\tau' + (\tau_k - \tau')$. In order to make pole k coincide with any spot Ψ , τ which we may choose on the surface of the reference sphere, the crystallite must be rotated so that its characteristic (001) pole takes a position Ψ' , τ' such that

$$\Psi' = \Psi - c_k
\tau' = \tau - C_k$$
(1)

Now, the probability that a crystallite will be so oriented that its characteristic (001) pole will find itself in an angular area $\delta\Psi$ long and $\delta\tau$ wide situated at the position Ψ' , τ' on the surface of the reference sphere is

$$P_{\stackrel{\Psi'}{\tau'},\stackrel{\Psi'}{\tau'}+\stackrel{\delta}{\delta}\stackrel{\Psi'}{\tau'}}=\underset{k}{S}f(\Psi')\bullet\Phi(\tau')\delta\Psi'\delta\tau' \qquad (2)$$

where the S means that the value of P is obtained by adding together the probability for each one of k crystallographically similar poles. Substituting Eq. (1) in Eq. (2) we have for the {110} poles,

$$P_{\Psi, \tau} = \underset{k}{S} f(\Psi - c_k) \cdot \Phi (\tau - C_k) \delta \Psi \delta \tau$$
 (3)

in which c_k and C_k are known for each pole of the $\{1\ 1\ 0\}$ plane-form and in which k assumes successive values from 1 to 6.

Since the number of poles, N, in the region marked out by Ψ , $\Psi + \delta\Psi$, τ , and $\tau + \delta\tau$ is proportional to the probability, we have

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$$N = K P_{\Psi, \tau}$$

Then the pole-density will be

$$D_{\Psi,\tau} = \frac{N}{A_{\delta\Psi,\delta\tau}} = K \frac{P_{\Psi,\tau}}{(g + \sin\tau) \delta\Psi\delta\tau}$$
(4)*

Substituting Eq. (3) in Eq. (4) we have

$$D_{\Psi, \tau} = K \frac{S f(\Psi - c_k) \cdot \Phi (\tau - C_k) \delta \Psi \delta \tau}{(g + \sin \tau) \delta \Psi \delta \tau}$$
 (5)

We must now choose some suitable specific type of function to represent $f(\Psi')$ and $\Phi(\tau')$. The function must be convergent, must decrease with increasing values of Ψ' and τ' , and must be symmetrical with respect to the zero reference position of Ψ and τ . Such a function may be expressed in many equivalent forms. Perhaps the most convenient form for our purpose is**

$$f(\Psi') = \sum_{i=1}^{i=n} \cos^{2i} a_i \Psi'$$

and

$$\emptyset (\tau') = \begin{cases} j = n \\ \sum_{i=1}^{\infty} \cos^{2j} b_i \tau' \end{cases}$$

When these distribution functions of the positions of the crystallites are used to predict positions of the {1 1 0} poles, they become

$$f(\Psi - c_k) = \sum_{i=1}^{i=n} \cos^{2i} a_i (\Psi - c_k)$$

$$i = 1$$

$$j = m$$

$$i = 1$$

$$(6)$$

$$(6)$$

Substituting equations (6) into equation (5) we have

$$D_{\Psi, \tau} = \frac{K \sum_{k=1}^{k=6} \{\Sigma_{1} \cos^{21} a_{i} (\Psi - c_{k})\} \{\Sigma_{J} \cos^{2J} b_{j} (\tau - C_{k}) \delta \Psi \delta \tau}{(g + \sin \tau) \delta \Psi \delta \tau}$$
(7)

^{*}g is a finite, but very small, quantity introduced to keep the fraction from becoming infinite for zero values of τ . For all except the smallest finite values of Ψ and τ its effect on the value of the fraction is negligible. The use of g is merely a method of correcting for other approximations.

^{**}The use of 1 instead of 0 for the lower limit of the summation merely alters slightly our scale of coordinates in plotting our final curves. It makes the calculations a little easier.

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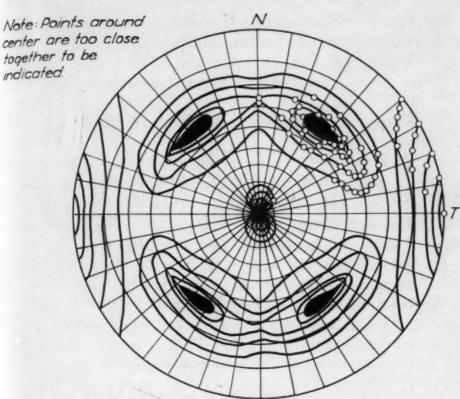


Fig. 8-Theoretical Pole-Figures Calculated From Equation (8).

Since we are only interested in the general shape of the calculated pole-figure, we shall assume that, if we confine the use of our equation to values of $(\Psi - c_k) = \pm 45^\circ$, and to values of $(\tau - C_k) = \pm 15^\circ$, the summation to i = 1 and j = 1 is a sufficient approximation.

Equation (7) therefore becomes

$$D_{\Psi,\tau} = \frac{K}{g + \sin \tau} \left\{ \left[\cos^2 a_1 \ \Psi \right] \left[\cos^2 b_1 \ \tau \right] + \left[\cos^2 a_1 \ (\Psi - 35) + \cos^2 a_1 \right] \right.$$

$$\left. (\Psi - 145) + \cos^2 a_1 \ (\Psi + 145) + \cos^2 a_1 \ (\Psi + 35) \right] \left[\cos^2 b_1 \ (\tau - 60) \right]$$

$$\left. + \left[\cos^2 a_1 \ (\Psi - 90) \right] \left[\cos^2 b_1 \ (\tau - 90) \right] \right\}$$

$$(8)$$

Equation (8) may be solved for values of Ψ and τ corresponding to a given value of $D_{\Psi,\tau}$ by the following method:

- (1) Substitute some value of τ into the equation.
- (2) Using systematically chosen values for Ψ , solve the equation for $D_{\Psi, \tau}^*$.

The exact values assigned to K, a, and b, are not important as they will only affect the scale of coordinates in the final graph. The ratio of b/a is, however, important and is known approximately from the diffraction data. This ratio has to do with the ratio of length to width of the "tear-drop" in the pole-figure. We have used the following arbitrary values in solving equation (8): K = 4, $b_1 = 4.5$, $a_1 = 1.5$. If we had used $b_1 = 9.0$ we should have had a still greater similarity between Figs. 5 and 8.

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- (3) Plot the values of $D_{\Psi, \tau}$ against the values chosen for Ψ . Label this line with the value assumed for τ .
- (4) Repeat operations (1), (2), and (3) for other systematically chosen values of τ , thus obtaining a chart such as is shown in Fig. 7.
- (5) Draw horizontal lines on the chart for systematically chosen values of $D_{\Psi,\tau}$.
- (6) The points where a given constant D line intersects the τ lines give the values of Ψ and τ which will yield the given constant pole-density on the reference sphere.
- (7) For each chosen value of $D_{\Psi, \tau}$, plot the corresponding values of Ψ and τ on a stereographic net (see Fig. 1c).
- (8) Connect the points for each value of $D_{\Psi,\tau}$ by a smooth curve. This gives a set of curves nested together, each curve corresponding to some definite degree of probability of orientation.

Each of these nested curves is the theoretical pole-figure for some per cent total reduction of cold-rolling. Fig. 8 is such a set of nested theoretical pole-figures. The circles represent values read off from Fig. 7. This gives one quadrant of the pole-figure. The other three quadrants were drawn by symmetry considerations and are therefore necessarily as correct as the one quadrant which was plotted. The agreement between the theoretical pole-figure of Fig. 8 and the experimental pole-figure of Fig. 5 for 90 per cent reduction is very striking. The theoretical figure even shows the "tear-drop" shape of the pole-figure for high per cent reductions. This striking agreement would seem to justify our assumptions as to the nature of the rolling process.

SUMMARY

We have made annealed, randomly oriented ribbons of very pure iron and of very homogeneous pure iron-nickel alloys up to about 22 per cent nickel. We have cold-rolled these ribbons under various controlled conditions and have examined the material for preferred orientation by X-ray diffraction methods. We have found that, below a very thin surface skin, the general type of preferred orientation is the same for all our rolled samples, and that the degree of preferred orientation depends only upon the per cent total reduction. We have made certain very simple assumptions as to the nature of the rolling process for body-centered cubic materials and have

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used these assumptions in developing equations which are capable of predicting the experimental pole-figures of our cold-rolled specimens.

ACKNOWLEDGMENTS

It is a pleasure to express our gratitude to the National Research Council Committee on Grants-in-Aid for making possible the purchase of certain equipment essential to our experiments. Without this timely aid in the early stages of this work it is very doubtful if it could have been carried to its present stage. It is a pleasure, too, to thank S. D. Smith and C. W. Siller for aid in preparing specimens and in constructing apparatus.

DISCUSSION

Written Discussion: By M. Gensamer and C. S. Barrett, Carnegie Institute of Technology, Pittsburgh.

The authors are to be congratulated for their ingenuity and patience in carrying through a painstaking study of the way in which we may represent the preferred orientations existing in a metal by simple scatter about an ideal texture, but there are certain of their conclusions which we would like to criticize.

The mathematical treatment forming the theoretical portion of this paper is based on three assumptions, and because the treatment gives a series of pole figures similar to those actually observed, the authors conclude that the three assumptions are justified and that the nature of the rolling process is satisfactorily described by them. The immediate object of the assumptions was to enable the authors to express the amount by which the orientation of crystallites differs from the ideal orientation by using only two quantities, Ψ and τ , representing rotation about two axes. Now any orientation of a crystal can be obtained from the ideal one by proper rotation about only two axes, so we believe that the authors' third assumption is unnecessary for their purpose. It follows, furthermore, that the authors' first and second assumptions are also open to criticism for a similar reason. It is possible to describe the orientation of any crystallite in a rolled structure by means of rotation about two axes, and the axes need not be those assumed here; that is, they need not be the face diagonals of a crystal in the ideal orientation. If, then, the axes of rotation can be chosen more or less at random, why should the authors attach any crystallographic significance to those two axes which they choose for purposes of mathematical convenience? It appears to us that any significance must lie not in the crystallographic index of the directions they happened to choose, but in the position of their axes relative to the direction of flow of material in the rolling process. In other words, we would attach importance to the symmetry of the pole figures with respect to the macroscopic flow instead of the symmetry with respect to crystallographic axes in a grain.

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Schmid and Boas,¹ elaborating on Wever's² explanation of the origin of preferred orientations in rolling, have shown that the type of preferred orientations developed can be predicted from the macroscopic dimensional changes, basing their whole argument on the known behavior of single crystals in tension and compression. In tension, single crystals rotate so that the slip direction (in iron the [111] direction) rotates directly toward the tension axis. In compression, the normal to the slip plane (in iron several planes may operate)³ rotates directly toward the compression direction. By properly combining tension and compression deformations to account for the dimensional changes in rolling, Schmid and Boas predicted the equilibrium orientations achieved by such rotations, and showed them to be in agreement with the observed preferred orientations. Now none of the rotations used was about crystallographically determined axes. It is on this ground that we question the authors' assumptions involving crystallographically determined rotation axes.

An experimental difference exists between the authors' pole figure and the pole figures derived for iron by Kurdjumow and Sachs⁴ and by Gensamer and Mehl.⁵ This difference lies in the failure of the authors to detect the compression component of the rolling texture. If Fig. 5 in this paper indicates all of the orientations of the sheet with respect to the X-ray beam for which X-ray photograms were prepared (shown by the dashed lines), the authors may well have missed the stereographic regions exhibiting the compression component because they failed to take a sufficient number of photograms. Their analysis may still apply to the tension component, but the compression component remains untreated.

There seems to be some confusion in the paper about the angle τ since its use in the equation on page 1088 is not consistent with the introduction to that equation. We also wish to inquire about the term $\sin \tau$ that appears in equations 4, 5, 7 and 8, for we suspect the term is introduced in an attempt to account for projection of the reference sphere upon the projection plane. If this is the case the term is incorrect for the argument applies to the reference sphere and not to a projection of the sphere.

On page 1099 of the paper the authors comment on the fact that the preferred orientations never achieve perfection. We agree that this is probably so, but we think the argument based on Fig. 4 is fallacious. No graph of this nature should plot any property against the per cent reduction of area if we are concerned with behavior beyond 90 per cent reduction. We should use Ludvik effective deformation, which is $\ln (1 + \lambda)$, when λ is the elongation calculated from the reduction of area.

One more point. The authors have chosen as their projection plane the T-N plane, as has Wever, instead of the R-T plane, as did Kurdjumow and Sachs. We think it desirable to have some standardization in this respect, since most metallurgists are not intimately familiar with the stereographic

¹W. Boas and E. Schmid, Z. tech. Physik, Vol. 12, No. 2, 1931, p. 71-75.

²F. Wever, Transactions, American Institute of Mining and Metallurgical Engineers, Institute of Metals Division, 1931, p. 52.

^aC. S. Barrett, G. Ansel, and R. F. Mehl, "Slip, Twinning and Cleavage in Iron and Silicon Ferrite," Transactions, American Society for Metals, Vol. 25, 1937, p. 702.

⁴G. Kurdjumow and G. Sachs, Z. f. Physik, Vol. 62, No. 9 and 10, 1930, p. 592-599.

⁵M. Gensamer and R. F. Mehl, Transactions, American Institute of Mining and Metallurgical Engineers, Iron and Steel Division, 1935, p. 277.

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p. 702. 30, p. 592-599. ang and Metalmethod. May we ask the authors for their reason for this choice? If one of these two conventions has some advantage, we think future workers in this field should adhere to it.

Oral Discussion

Reply by Professor Davey: I am very glad that Drs. Gensamer and Barrett have found the error in the equations on pages 1088 and 1089 of the paper. In both equations they should read $(90^{\circ} - \tau)$. In our first drawing of Fig. 2, the angle which is now labeled $(90 - \tau)$ was labeled τ . The designation of the angle was changed as a convenience in the theoretical discussion, and the corresponding changes on pages 1088 and 1089 were overlooked. Fortunately these equations do not enter directly into the theoretical discussion so that the validity of that discussion is not impaired. The use of $\sin \tau$ in our Eqs. 4, 5, 7, and 8 is made clear by reference to our Fig. 2.

I am afraid I cannot agree with the criticism of our three basic assumptions on which the theoretical part of the paper is based. It is true that the pole of any crystal plane can be made to assume any desired orientation by rotating the crystal about any two randomly chosen axes; but because of the possibility of rotating the crystal plane about its own pole as an axis, it is not possible to obtain any desired orientation of a crystal by merely rotating the crystal about two randomly chosen axes. We chose the face-diagonals of the unit-crystals as axes of rotation because the diffraction patterns showed clearly that such axes could be so used. It may be possible to find other axes which will do as well, and which will explain the data as simply, but if so we have so far been unable to find them.

Drs. Gensamer and Barrett refer to an interpretation of orientation in terms of the macroscopic flow of metal. With this, we have no quarrel. It is always possible to consider a given experimental fact from more than one point of view. Since experiment shows that orientation of crystallites occurs as a result of rolling, it must be possible to imagine a sequence of motions of rotation which will account for the observed end result. This we have tried to do. The reasons underlying these supposed motions must certainly be related to the crystal structure of the rolled material, and to the forces of compression and tension set up by rolling. In this paper we were interested only in the probable motions of rotation of the crystallites, not in the reasons for those motions.

In our short discussion of Fig. 4, we were of course acquainted with the theory of elastic deformation. Since, however, we were interested solely in motions of rotation as a method of arriving at a preferred orientation, we merely called attention to the experimental fact that complete orientation was not found, and to the obvious conclusion from the experimental data, that complete orientation can never be obtained by rolling. We realize of course that such a conclusion, based on direct experiment, is consistent with well accepted theory.

The present work represents orientation data taken from about 900 different diffraction patterns. These were taken on 53 specimens at various depths. It would have been much too big a job to have taken so many patterns that the whole pole-figure was made up of adjacent experimental points. We believe,

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however, that the number of experimental points on one pole-figure compares favorably with those found in most published pole-figures. It should be noted too, that we have shown the experimental limits for the location of our poles, and not merely a set of points around which a pole-figure may be drawn by theory. In all scientific work it is assumed that a smooth curve can be drawn between experimental points spaced reasonably close together. In a few instances, notably in work on surface tension, this common practice has led to serious error. The assumption is, however, necessary if we are ever to make graphic representation of data. In the present work, we have presented Fig. 5 as a typical sample of the pole-figures which we have found. We are encouraged in this by the fact that all our other pole-figures (in which no especial effort was made to use standardized settings of our specimens), are practically indistinguishable from Fig. 5.

The method of preparation of the specimen was such that we had every chance to get rid of our oxygen and carbon in specimens. Before they were rolled they were heat treated for a long time up to as close to the melting point as we dared go. It is well known that with such treatment carbon and oxygen will be given every chance to get to the surface and combine with the hydrogen. The hydrogen which we used was very free from nitrogen. Of course we realize that our specimens were full of hydrogen. Tests so far indicate that hydrogen does not effect orientation.

Originally our pole-figures were made in the R-T plane. We made new ones in the T-N plane in order that our figures might be compared more easily with the well-known work of Wever. We should be happy to standardize on any projection plane recommended by the American Society for Metals. Until such official recommendation is made, however, we believe in following the lead offered by previous literature in order to avoid confusion as much as possible.

Additional Written Discussion: By M. Gensamer and C. S. Barrett following W. P. Davey's oral discussion.

We do not deny that the authors have chosen a set of axes that are useful and convenient for describing their pole-figure. But we insist that there are an infinite number of pairs of axes that would also rotate the "ideal orientation" into any desired orientation. In the accompanying stereographic projection we show some of these. Two {110} poles which define the ideal orientation are shown at A and A' in the figure. These poles have been rotated into positions corresponding to a certain crystallite in the rolled structure chosen at random and represented by B and B'; that is, A has been rotated to B simultaneously with the rotation of A' to B', and this rotation has been carried out four different ways, using four different sets of axes, namely axes 1 and 1, 2 and 2, 3 and 3, 4 and 4. The lines connecting A with B and A' with B' show the paths of the poles when the crystal is rotated first about one axis of a pair, then about the other. There are clearly an infinite number of other pairs of axes which will accomplish the same net result as the four pairs we have indicated. The authors' choice of one set out of this infinite number is, therefore, an arbitrary choice and we cannot agree that the authors have shown any connection between the axes which they have found convenient and the underlying mechanism of crystal rotation.

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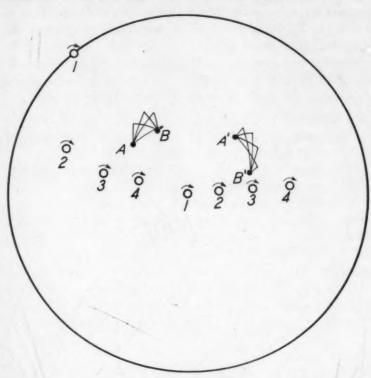
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We feel that the evidence for the existence of what Wever called the compression component of the texture is very good and cannot be ignored. If Dr. Davey does not find it, there is a disagreement which must be looked into, perhaps by the exchange of samples.

Authors' Closure

Drs. Gensamer and Barrett are quite correct that an infinite number of pairs of axes can be used to transfer the poles A and A' in their stereographic projection to new location at B and B'. But to express the orientation of a crystal plane in space we need only the location of its pole on the sphere of reference but also the amount by which the crystal plane has been rotated using its pole as an axis. I think I am correct that each of Gensamer and Barrett's four pairs of axes will give the crystals different orientations about their poles by the time the poles themselves reach B and B'.

In our experiments we started with random orientations of crystallites, random not only with respect to the location of their {110} poles but also with respect to the orientation of each of the {110} planes about its own pole. We ended up with a preferred range of positions for the {110} poles themselves and with a preferred range of orientations about those poles. The orientations

⁶F. Wever, *Transactions*, American Institute of Mining and Metallurgical Engineers, Institute of Metals Division, 1931, p. 52.

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about the {110} poles in the center and in the "tear-drop" portions of our Fig. 5 may be found easily by means of the poles recorded at the extreme right and left of the figure and vice versa. Our three basic assumptions in the theoretical part of our paper enable us to get from a completely random orientation to our experimentally found orientation not merely in terms of the location of the {110} poles but also in terms of the correct asimuth with respect to rotation about those poles.

The differences between our results and those of Kurdjumow and Sachs and of Gensamer and Mehl may possibly have to do with matters of technique in the preparation of the metal or in the rolling process or both.

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THE APPLICATION OF X-RAY DIFFRACTION TO THE STUDY OF FATIGUE IN METALS

By CHARLES S. BARRETT

Abstract

In an attempt to evaluate the usefulness of X-rays in the study of fatigue, the papers on this controversial question were critically reviewed, the co-operation of the A.S.T.M. committees on X-rays and Fatigue was secured, and some 200 X-ray diffraction patterns were prepared. The X-ray patterns undergo changes both above and below the endurance limit, the changes being similar in type at all stress levels and caused by plastic deformation of the grains. Therefore, the detection of these changes is not necessarily a sign of impending fatigue failure. However, since the magnitude of these changes decreases with decreasing stress level, some practical applications to the detection of progressive damage appear to be possible. X-rays are useful in determining the depth to which a metal is cold-worked during the preparation of a fatigue specimen, a depth of about 0.002 to 0.008 inch in common materials.

INTRODUCTION

It would be of considerable importance if X-ray diffraction could be used as a nondestructive test to follow the progressive deterioration of materials under repeated stressing and to predict impending failure from fatigue. Yet in spite of the practical and scientific interest in fatigue there have been relatively few X-ray studies in the field, and these have been too incomplete to determine the possibilities and limitations of X-ray methods.

The first experiments were those of Dehlinger, who investigated the changes in cold-rolled copper and silver sheet when subjected to repeated bending. He found that the diffraction lines, which were quite broad with the cold-rolled material, became definitely sharper

¹U. Dehlinger, Naturwissenschaften, Vol. 17, 1929, p. 545, and Metallwirtschaft, Vol. 10, 1931, p. 26-28.

A paper presented before the Eighteenth Annual Convention of the Society held in Cleveland, October 19 to 23, 1936. The author, Charles S. Barrett, is a member of the Metals Research Laboratory and Lecturer, Department of Metallurgy, Carnegie Institute of Technology, Pittsburgh. Manuscript received June 24, 1936.

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as a result of the fatigue stressing, and also that spots appeared in the otherwise uniformly darkened diffraction lines. ascribed these changes to recovery and recrystallization processes operating at room temperature during the stressing. The changes were found only after bending had continued nearly to the point of fracture, and then only in photograms taken of portions of the metal that had been highly stressed. As they were not found at stresses below the endurance limit of the material, Dehlinger concluded that plastic flow did not occur in this stress range. Dehlinger's experiments, however, are hardly conclusive on this point, for slip lines have been observed to form and strain hardening to occur as a result of fatigue under a safe range of applied stress.2 Pfarr3 has also observed the sharpening of diffraction lines from a hardened steel as a result of 4 x 10° reversals of stress of an unspecified amplitude, and H. A. Smith4 was led to suspect a recrystallization effect at the surface of fatigue breaks in aluminum wires. McCutcheon,⁵ on the other hand, concluded that the relatively sharp diffraction lines from 0.10 per cent carbon steel in the annealed condition became sharper only as a result of fatigue at stresses below 10,000 pounds per square inch and became widened by stressing above this range.

Diffraction patterns of the Laue type published by Stephen and Jones⁶ revealed severe distortion of the grains in broken fatigue specimens of annealed steel, the distortion being recognized in the photograms as "asterism" (distortion of Laue spots). However, the photograms were not made in such a way as to differentiate between deformation occurring at the instant of final failure and any progressive deformation that might have occurred previously, for the samples were X-rayed only at points directly beneath the rough portion of the broken surface. Gough has also found asterism in a Laue photogram in the case of repeated stressing of a single crystal of aluminum, and Fahrenhorst and Ekstein with a crystal of cadmium. Neither of these Laue studies has revealed the dependence of asterism on stress amplitude or number of cycles.

²H. J. Gough. Edgar Marburg Lecture, *Proceedings*, American Society for Testing Materials, Vol. 33, II, 1933, and references included therein.

^aB. Pfarr, *Zeit. Tech. Phys.*. Vol. 14, 1933, p. 220.

F. Wever and B. Pfarr, *Mitt. Kaiser Wilhelm Inst. für Eisenforschung*, Vol. 15, 1933, p. 497 1933, p. 497

⁴H. A. Smith, Physics, Vol. 5, 1934, p. 412.

⁸D. M. McCutcheon, Metals and Alloys, Vol. 5, 1934, p. 230.

eR. A. Stephen and W. R. D. Jones, Metallurgist, supplement to Engineer, June 30,

⁷H. J. Gough. loc. cit. W. Fahrenhorst and H. Ekstein, Zeit. f. Metallkunde, Vol. 25, 1933, p. 306-308.

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If the experiments mentioned thus far are considered in the light of other fatigue researches they are not as contradictory as they appear at first sight. With internally stressed materials, fatigue has been shown to bring about a reduction of internal stresses of the homogeneous, macroscopic type8 and would be expected, therefore, to reduce also the stresses of the microscopic type which are responsible for line broadening. This might result either from plastic movement of the material or from recovery (with or without recrystallization) during the stressing. With previously unstressed material, on the other hand, the plastic flow that occurs during fatigue must result in distorted grains, asterism in Laue photograms, and widened diffraction lines. From this point of view the studies reviewed above are mutually consistent with the exception of the observation of line sharpening in steel at very low stress levels, and this is an exceedingly small effect, as will be seen from McCutcheon's published photograms.

The X-ray lines from CuAl₂ have been noted in photograms of a long used duralumin propeller blade and, because of their variation in intensity throughout the blade, have been ascribed to precipitation accelerated by the fatigue process.9 Kempf, 10 however, pointed out that these X-ray patterns did not differentiate between precipitated CuAl₂ and primary CuAl₂, and that the latter is always present in commercial heat treated forgings of 25 S-T such as are used for propeller blades. He reported that while the metallurgical microscope is able to distinguish primary from precipitated CuAl, and is more sensitive in detecting the initial stages of precipitation than is X-ray diffraction, it has failed to reveal any precipitation in this material resulting from large numbers of stress reversals either above or below the endurance limit, and that mechanical properties have also failed as an inspection method for following progressive deterioration.

A number of papers on the applicability of X-rays to fatigue have come from the laboratory of F. Regler in Vienna and have caused considerable controversy. These papers describe a back reflection camera of new design in which the film is conical in shape¹¹

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306-308.

⁸H. Buchholtz and H. Buhler, Archiv für das Eisenhüttenwesen, Vol. 6, 1933, p. 335-340; Stahl und Eisen, Vol. 53, 1933, p. 1330-1332.

⁸G. L. Clark and H. A. Smith, Physical Review, Vol. 43, 1933, p. 305. G. L. Clark, Electrical Engineering, Jan. 1935, p. 3-14. ¹⁰L. W. Kempf, Physical Review, Vol. 43, 1933, p. 942-943.

¹¹F. Regler, Zeit. für Phys., Vol. 74, 1932, p. 547-564; Mitteilungen des Technischen Versuchsamtes, Wien, Vol. XX, 1931; Ergebnisse der Technischen Röntgenkunde III, 184-193, Leipzig (1933).

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and discuss the application of this camera to the measurement of stress and fatigue in metals. In this series of publications Regler at first claimed remarkable accuracy in the measurement of stress by averaging the widths of the individual spots in a Debye ring, the width being measured in the peripheral direction (tangential to the ring).12 In his earlier papers he placed the probable error in his stress measurement at \pm 0.1 kilograms per square millimeter (142 pounds per square inch) as a result of an accuracy of ± 0.001 millimeter in the determination of average spot width for 30 chosen spots which vary in width from 0.22 to 0.65 millimeter. Critics have objected to his claim of this accuracy for the average of individual readings so divergent,13 and he himself in his later papers altered his position and characterized the method as unsuited for quantitative work on many commercial materials.14

A second method much used by Regler is based on the radial width of the diffraction rings. 15 However, instead of measuring the width by preparing a microphotometer trace and measuring the width between the points of half maximum intensity, as is usually done, Regler measures the separation between two faint sharp lines or striae which he believes form the inside and outside boundary of the diffraction rings whenever the camera and X-ray tube are properly adjusted.16 He states that an experienced eye looking at a properly illuminated and magnified film can detect these faint sharp edges, although only with difficulty, and that they can be measured to ± 0.002 millimeter, corresponding to an accuracy of stress measurement of ± 0.1 kilogram per square millimeter (142 pounds per square inch). Regler also states that the radial line width at a fracture is independent of the previous heat treatment and of the stress conditions causing the fracture whether in static loading or in fatigue, and is only dependent on the composition of the material. Consequently, he concludes that the life of a stressed member may be

¹²F. Regler, Mitteilungen des Technischen Versuchsamtes, Wien, XXI, 1932; Ergebnisse der Technischen Röntgenkunde, Leipzig, III, 184-193, 1933; Phys. Zeit., Vol. 33, 1932,

F. Regler and F. Lihl, Zeit. Phys., Vol. 89, 1934, p. 537-545.

E. Wever and H. Möller, Archiv f. d. Eisenhüttenwesen, Vol. 9, 1935-36, p. 47-55. ¹⁴F. Wever and H. Möller, Archiv f. d. Eisenhüttenwesen, Vol. 9, 1935-36, p. 47-55 and Regler's discussion thereto, p. 52-55.

¹⁵References to the previous literature on this subject have been reviewed by the author in Metals and Alloys, Vol. 5, August 1934, p. 170-174.

¹⁶F. Regler, Mitteilungen des Technischen Versuchsamtes, Wien, XX, (1931); XXI, (1932); XXII, (1933); XXIII, (1934); Ergebnisse der Technischen Röntgenkunde III (1933); Berichte der 2ten Internat. Schienentagung, Zurich, 1932; Ann. der Phys., (5), Vol. 19, 1934, p. 637-664; Schweizer Bauzeitung, Vol. 105, Jan. 1935.

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32: Ergebnisse Vol. 33, 1932,

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predicted by noting how close and at what rate the radial width is approaching the limiting value that corresponds to fracture.

It was to be expected that the importance of these conclusions to engineers would arouse interest in other laboratories. and Scheu,17 also in Vienna, were the first to make a careful trial of Regler's method and concluded, contrary to Regler, that there were no sharp boundaries for Debye lines; that the width of the lines at half maximum was not independent of previous thermal and stress history, but varied widely; and that the widening that results from static stressing below the yield point is negligible. A recent investigation of Regler's work by Wever and others under the auspices of the Verein deutscher Eisenhüttenleute resulted again in an adverse report, 18 in which it is stated that all efforts in other laboratories to find measurable sharp boundaries to the Debye lines and to use the Regler method have failed; that all tests of the method in Regler's laboratory in the presence of witnesses failed when the identity of the specimens was concealed from Regler; that Regler refused to demonstrate the method in Germany; and that finally Regler refused to co-operate further in rigorous tests of the method in his own laboratory, stating that he feared overstraining his eyes. While there is a considerable amount of personal animosity apparent in these critical studies of Regler's method, the criticisms do not appear to have been refuted, and Regler's claims therefore remain unsubstantiated at the present time.

The purpose of the investigation reported herewith is to evaluate the practical usefulness of X-rays in the fatigue field: to determine whether or not changes in X-ray diffraction patterns can be taken as definite evidence that fatigue failure is subsequently to occur, and if not, to learn whether or not they can be correlated in any way with stress intensity; to compare the relative sensitivity of different X-ray methods to such changes, and to compare different ways of quantitatively measuring the diffraction patterns; to evaluate the possibility of an accelerated fatigue test using a single specimen, a small number of cycles at various stress levels, and a series of X-ray diffraction photograms as an indicator; and to investigate X-ray diffraction as a tool for determining the distribution of damaged material in fatigue specimens containing stress concentrations and also for measuring the depth of cold work from machining fatigue specimens.

 ¹⁷P. Ludwik and R. Scheu, Metalkvirtschaft, Vol. 13, 1934, p. 257-261; see also Ludwik and Scheu, and Regler, Metalkvirtschaft, Vol. 13, 1934, p. 427-429.
 ¹⁸F. Wever and H. Möller, Archiv f. d. Eisenhüttenwesen, Vol. 9, 1935-36, p. 47-55.

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WIDENING OF DIFFRACTION LINES BY FATIGUE

Technique—In order to have the greatest sensitivity to the widening of lines all exposures were made in the back reflection type of camera in which the angle between incident and diffracted rays is nearly 180 degrees. The camera used had a flat film which was rotated about the incident beam as an axis during the exposure in order to avoid spottiness in the diffraction lines.* A shield was provided so that a series of exposures (up to five in number) could be made on the same film under identical conditions without overlapping.

The surface layers which had been cold-worked in machining the specimens were always removed by etching. Unstressed specimens were used to give diffraction lines of standard width, to which others could be compared.

A. Structural Silicon Steel

Material—Rotating beam fatigue samples were available of a silicon steel intended primarily for use in structural members, with endurance properties as shown in Fig. 1.**

Results—All diffraction lines from the unetched surfaces of fatigue breaks in these specimens were very broad (The Ka doublet was not resolved), but no measurements of the exact widths were made.

The outer surface of a specimen that had broken after 873,000 cycles at 52,000 pounds per square inch was X-rayed in the neighborhood of the break (at an area from 1 to 3 millimeters from the break). The lines were broadened, though less so than those from the broken surface where Ka doublet was no longer resolved. It is possible that this resulted in part from deformation at the instant the specimen broke.

Diffraction lines from a specimen that had undergone 81,490,300

^{*}The camera is described elsewhere: C. S. Barrett, Metals and Alloys, Vol. 4, 1933, p. 63, and the essential dimensions in this work were as follows: two pinholes of 1 millimeter diameter, separated 5 to 6 centimeters, collimated the X-ray beam, which passed through a hole in the film 1 millimeter from the second pinhole and irradiated a spot approximately 2 millimeters in diameter on the specimen which was 100 millimeters \pm 1 from the film Cobalt Ka_1 and Ka_2 radiation was reflected from (3 1 0) planes of the steel specimens, and copper, Ka and Ka_2 radiation was reflected from (5 1 1) planes of aluminum alloys.

^{**}The steel analyzed C 0.36, Mn 0.77, Si 0.34, P 0.047, S 0.040 per cent (meeting A.S.T.M. Standard Specification A94-33) and had the following mechanical properties: endurance limit 47,000 pounds per square inch, tensile strength 93,400, yield point 66,700, elongation in 2 inches 1.9 per cent, reduction in area 57.8 per cent. The samples for this test and for the other tests in this paper had been stressed one or two years previous to the X-ray study, but it was felt that this time interval would not adversely affect the results, and none of the results has altered this opinion.

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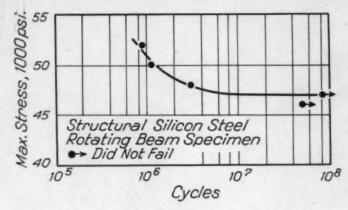
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r cent (meeting ical properties: ld point 66,700, samples for this ears previous to reversals of stress at 47,000 pounds per square inch (just under the endurance limit) without failing were of approximately the same width as the lines from the same material unstressed (see Fig. 2a and 2b). There is a suggestion of a slight widening from the fatigue



-Fatigue Characteristics of the Structural

stressing, but the magnitude of the effect is so small that visual observation can scarcely detect it, and microphotometer measurements would be necessary to prove its existence.

B. Aluminum Alloy, 25 S-T

Material—To avoid the variations of line width over the surface of a specimen that might be expected in any high strength aluminum alloy, it was decided to use an annealed alloy. After consideration of the fatigue (or S-N) curves of various alloys, 25 S-T was chosen as the most suitable material available in the form of previously stressed rotating beam specimens.*

Results for 25 S-T-Eleven exposures were made at various stress levels both above and below the fatigue limit. As far as can be judged by the eye in no case did the stressing alter the line width. If any such effect exists, it is so small as to escape detection without careful microphotometer measurement of the films, and in any case it is too small to offer any hope of a practical method of following the progress of fatigue or of predicting impending fatigue failure. One series of exposures is reproduced in Fig. 3.

^{*}The composition was Cu 4.50, Fe 0.37, Si 0.70, Mg 0.80%, balance Al; after forging and rolling the material was held at 960 degrees Fahr. for 10 hours, quenched in water aged at 340 degrees for 12 hours and given a degrowthing treatment at 440 degrees for 24 hours. The properties of the slab in the transverse direction (the direction in which the fatigue specimens were cut) were as follows: tensile strength 46,600, yield strength 26,400, endurance limit 13,000.

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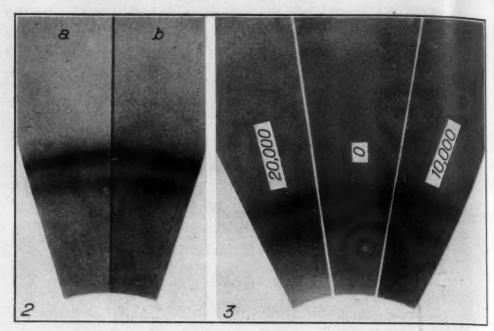


Fig. 2—Diffraction Lines From Silicon Structural Steel. (a) From Unstressed Material. (b) From Area Stressed 81,490,300 Cycles at the Endurance Limit (47,000).

Fig. 3—Diffraction Lines From a Single Specimen of Aluminum Alloy 25 S-T. Segment Marked "0" from Unstressed Material. Segment Marked "20,000" from Area Stressed 627,000 Cycles at 20,000 Pounds Per Square Inch (Above the Endurance Limit). Segment Marked "10,000" from Area Stressed 627,000 Cycles at Approximately 10,000 Pounds Per Square Inch (Below the Endurance Limit).

Exposures that were made of the unetched broken surface gave lines of practically the same width whether the beam struck the smooth portion of the broken surface or the rough portion. It must be concluded from these studies that under these conditions cold work does not appreciably widen lines in this material.

This is the usual result in X-ray studies of cold work in aluminum, although in a few cases line widening has been observed.

C. Conclusions on the Line Width Method

The radial widening of Debye lines by fatigue stressing is so small that a testing method based on it must involve careful measurements with a microphotometer. This fact, together with the fact that in some materials the effect appears to be entirely absent, makes the line width method unsuitable for extensive practical applications.

DISTORTION OF DIFFRACTION SPOTS BY FATIGUE

Early in this investigation it became apparent that a more sensitive method than the one discussed above is to use a stationary speci-

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men and film, which provides a pattern of diffraction spots, and to observe the amount of blurring and distortion of these spots. (The phenomenon is frequently called "asterism" and is equivalent to "peripheral widening" if the spots are made by monochromatic radiation.)

In this method each grain reflects X-rays to form one or more definite spots on the film. When the grains are undistorted the

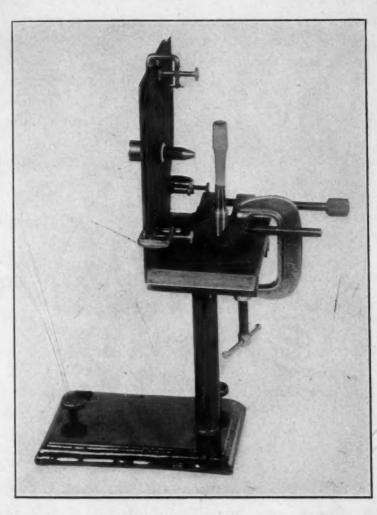


Fig. 4—Simple Back Reflection Camera for Studying the Distortion of Diffraction Spots by Fatigue.

atomic planes produce sharp spots, while bending or distortion of the grains will lead to elongated spots and diffuse spots. The prominence of the blurring is a semi-quantitative indication of the amount of cold work the specimen has received.

Technique—This method has been widely used in various forms:

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"monochromatic pinhole photograms" and "transmission Laue photograms" with X-rays passing through thin sheets of metal; reflection of X-rays in the forward direction when incident at small angles on a metal surface, and back reflection photograms, with X-rays falling perpendicularly on the metal and reflecting back at high angles. It was soon concluded that the back reflection camera was most satisfactory for the present problem, and this was used throughout the work reported herein. Radiation from a cobalt target was used for steel and for copper specimens, and from a copper target for aluminum specimens. A few exposures were made on the precision camera used for line-width studies, but most of the work was done on the simple camera of Fig. 4. In this camera the specimen was 4 centimeters from the film. The beam was defined by a pinhole 1 millimeter in diameter, 4 centimeters long, extending through the film and to within 1½ centimeters of the specimen.

The surface layers, which had been cold-worked in machining the specimens, were removed by etching,* and photograms of the material in the unstressed condition were always prepared for comparison with the stressed material. Care was taken to have the X-ray beam fill the pinhole system as completely as possible in each exposure, thus keeping the diameter and divergence of the beam constant, and an attempt was made to get the same blackening of the film throughout a series of related exposures.

A. 0.19 Per Cent Carbon Steel

Material—Tensile and fatigue data were known for a series of fatigue specimens of annealed steel containing 0.19 per cent carbon. The S-N curve is given in Fig. 5. The steel analyzed C 0.19, Mn 0.44, Si 0.03, S 0.035, P 0.011. The properties were as follows (after a 2-hour anneal at 875 degrees Cent.): endurance limit with 0.273-inch diameter specimen 27,800, proportional limit 28,500, yield point 29,750, tensile strength 55,500, reduction of area 66.3 per cent, elongation 44.5 per cent. The specimens had been stressed one to three years prior to the X-ray tests.

^{*}Steel specimens were etched with 50% nitric acid, precautions being taken to get a uniform attack. Aluminum alloys were etched with reagent described by C. M. Tucker. Metals and Alloys, August 1930, page 655: cone. HCl 45%, cone. HNO₃ 15%, cone. HF 15%, warm water 25%. Copper was etched first in solution (A), cone. HNO₃ with excess CrO₃, for 5-10 seconds, then in solution (B), CuNH₃Cl, 10% solution in water, for 10 seconds, then washed and etched in solution (A) again until a smooth surface was obtained. Each cycle removed about 0.0005 inch and was repeated until the required depth was reached.

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Thirty X-ray exposures were made of this material to test the sensitivity of the method to changes during fatigue.

Results on previously stressed samples—A rotating beam specimen (R. R. Moore type) that had withstood 12,360,000 cycles at a maximum stress of 26,000 pounds per square inch was X-rayed at several places on the outer surface. Photograms made at stress levels of 26,000 and 23,000 showed a slight but definite blurring of spots. These stresses were below the endurance limit and yield

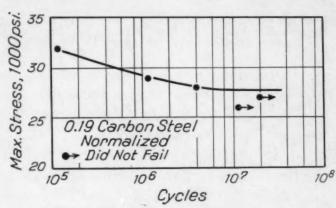


Fig. 5-Fatigue Characteristics of the 0.19 Per Cent Carbon Steel.

points for the material. One of these is reproduced in Fig. 6b, to be compared with the unstressed material of Fig. 6a. Distortion of spots did not appear, however, in a photogram at the 15,000 pounds per square inch level.

A cantilever specimen that had broken after 300,000 cycles at a stress level slightly above the endurance and proportional limits and the yield point produced photograms with diffuse spots. Four places at the maximum stress level were X-rayed; of these, the places located near the ends of fatigue cracks gave photograms in which all spots were distorted, while the others gave a few sharp spots mixed with a majority of diffuse spots, very similar to the photogram of Fig. 6c. As in the case reported in the preceding paragraph, photograms at very low stress levels showed no blurring of spots.

A study of the progress of fatigue—Two series of tests were run on R. R. Moore rotating beam machines to determine how the photograms changed during a fatigue run. The major difficulty in such a test is to obtain a specimen with the surface material in suitable condition: the surface must have a small grain size, be free from cold work, free from pits, and must be of the same composition

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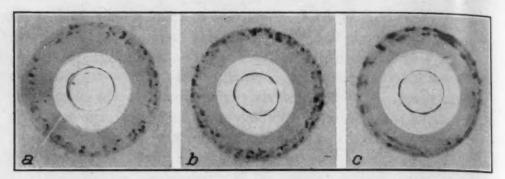


Fig. 6—Photograms of 0.19 Per Cent Carbon Steel. (a) Unstressed. (b) After 12,360,000 Cycles at 26,000 Pounds Per Square Inch. (c) After 341,000 Cycles at 29,000 Pounds Per Square Inch (Exposed 2 Millimeters From the Break).

as the interior. An attempt to meet these conditions was made by machining the specimen somewhat oversize and then removing successive layers by etching and polishing. Etching was done with a 10 per cent KI solution containing excess iodine.

A number of days were spent in attempting to find an etchant that would leave the surface free from microscopic etching pits. The above technique was the best one tried, but was far from satisfactory.

Layers 0.001 inch to 0.002 inch thick were removed by each etching and about 0.001 inch by each polishing. These alternate treatments, ending with an etch deep enough to remove the cold work from the last polishing operation, removed all metal disturbed by machining. The resulting surface was reasonably smooth except for fibering in the longitudinal direction. The grips were protected from attack by a wax coating.

In spite of precautions to keep pitting to a minimum and to keep a circular cross section, the specimens failed prematurely during the stressing. One specimen stressed at 29,000 pounds per square inch failed after 341,000 cycles, yet previous results on polished specimens of the same material indicated that it should have withstood five times this many cycles, or that it should have withstood this number of cycles at 31,000 pounds per square inch.

After preparing photograms of marked areas on the unstressed specimens, the first sample was given 310,000 cycles at 29,000 pounds per square inch, then removed from the machine and X-rayed immediately at several places on the surface. Stressing was resumed the following day, continuing until failure occurred (after a total of 341,000 cycles), and then further photograms were taken.

Results of first progressive experiment—At points of maximum stress (29,000), above the endurance, proportional and yield limits,

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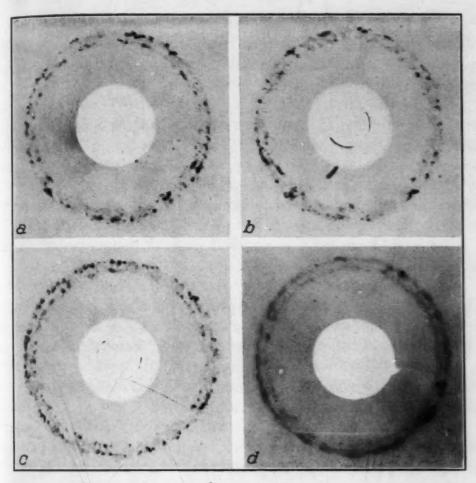


Fig. 7—Photograms of 0.19 Per Cent Carbon Steel, Second Progressive Experiment. (a) Unstressed. (b) After 300,000 Cycles at 25,000 Pounds Per Square Inch and 300,000 Cycles at 26,000. (c) After Stressing in (b) Had Been Followed by 220,000 Cycles at 27,000. Exposed at a Point Under 26/27 Maximum Stress. (d) Same as (c) Exposed Near the Break.

there was a considerable blurring of spots after 310,000 cycles, and a further blurring after 341,000 cycles (Fig. 6c). The photograms clearly showed that distortion of the metal was a maximum at the break and was somewhat less 2 millimeters from the edge of the break.

At points of lower stress some photograms (taken at a stress level of 27,000 pounds per square inch) were obtained in which occasional spots were diffuse, and others were obtained at still lower stresses (22,850), in which there was no change in the appearance of the spots. In view of the abnormal behavior of these specimens in fatigue it is difficult to relate these latter stresses to the effective endurance limit, but probably the points stressed at 27,000 were above the limit and those stressed at 22,850 were below it.

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The second specimen was used to test the possibility of using X-rays for an accelerated fatigue test. It was given runs of 300,000 cycles each at successively increasing stresses and examined between each run with X-rays.

Results of second progressive experiment—There was only a very slight blurring of spots after the first run of 300,000 cycles at 25,000 pounds per square inch. There appeared to be no further change when this was followed by 300,000 cycles at 26,000 and then by 220,000 cycles at 27,000 after which it failed. (In the latter exposure the X-ray beam was 2 millimeters from the break at its closest point.) Some typical films from this series are reproduced in Fig. 7.

As in all other cases, photograms made at the edge of the break showed severe distortion.

Conclusions from experiments on 0.19 per cent carbon steel— There is severe distortion of the grains at the surface of a fatigue break and at the ends of a fatigue crack. In areas slightly removed from these places the grains are less distorted. The distortion is equal at all places of equal stress and decreases with decreasing stress down to stresses below the yield point and endurance limit, provided the specimen has received a large number of cycles.

On the basis of these experiments it appears useless to attempt to develop an accelerated fatigue test using a single specimen, a small number of cycles, and a series of X-ray photograms to follow the effects of stressing. From the whole series of 0.19 per cent carbon experiments it appears that there is no abrupt change in the spot patterns with increasing stress as the stress passes from the safe to the unsafe range. Furthermore, the X-ray photograms in one case did not change progressively during the stressing even though the specimen was approaching failure.

B. Silicon Structural Steel (0.36 Per Cent Carbon, 0.34 Per Cent Silicon)

The specimens were the same ones that have been described in the section IIA on Widening of Diffraction Lines and were of the rotating beam type, previously stressed. The endurance limit was 47,000 pounds per square inch (Fig. 1).

Results—A specimen that had been broken by 873,800 cycles at a maximum stress of 52,000 pounds per square inch was X-rayed in a number of places on the surface, corresponding to different

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stress levels, with the results given in Table I. None of these stresses was over the yield point, and only the first was over the endurance limit. A photogram of the broken surface was more blurred than any of this series.

A specimen that had been run for 81,490,300 cycles at the endurance limit, 47,000 pounds per square inch, without breaking was studied at various places on the surface. Areas at the same stress level gave similar photograms. Areas stressed at 47,000 gave diffraction spots that were diffuse compared with areas stressed at 27,000 or 21,000 (Fig. 8).

Conclusions, structural silicon steel—As in the case of 0.19 per cent carbon steel, the distortion of grains is a maximum at the fatigue break and decreases with decreasing stress level. It is still appreciable at stresses well below the endurance limit after several hundred thousand reversals, and probably does not disappear until the stress drops below about half the endurance limit stress.

Comparing these results with those obtained on the same material by the line-width method it is clear that the effects of fatigue stressing on this steel can be more easily recognized in the blurring of spots than in the broadening of lines.

C. 0.42 Per Cent Carbon Steel with Stress Concentration

The sensitivity of the method was tested with a medium carbon steel (0.42 per cent carbon) in the form of a cantilever specimen containing a transverse hole which produced a concentration of stresses. The steel was a "clean" steel prepared by C. H. Herty, Jr., with an endurance limit of 33,800, a yield strength of 37,200 and an ultimate strength of 73,800 in the as received, hot-rolled condition. It had been stressed three years previous to the X-ray test.

A hole 1/4 inch diameter had been drilled and reamed diametrically through the tapered specimen at the section where the diameter

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was 1 inch, and the specimen had been given 11,699,000 cycles at an average nominal stress of 21,000 pounds per square inch (figured as the bending moment divided by the net section modulus). Under these conditions at the lateral edge of the hole the theoretical stress concentration factor of 1.96 predicted a maximum stress of 41,000.

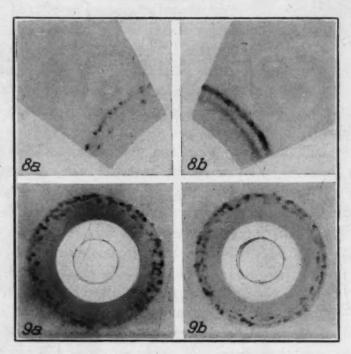


Fig. 8—Photograms of Structural Silicon Steel. (a) After 87,490,300 Cycles at 21,000 Pounds Per Square Inch. (b) After 81,490,300 Cycles at 47,000 Pounds Per Square Inch.

Fig. 9—Photograms of Spring Steel, 0.85 Per Cent Carbon. (a) Unstressed. (b) After 18,000,000 Cycles at 33,500 Pounds Per Square Inch.

well above the normal fatigue strength (33,800) and yield strength (37,200) of the material. On the basis of the fatigue stress concentration factor, however, the lateral edge of the hole was stressed slightly below the endurance limit.

The fatigue stress concentration factor was obtained from the re-

$$k = \frac{\text{normal endurance limit}}{\text{endurance limit with hole}} = \frac{33,800}{21,800} = 1.55.$$

It was thought that X-ray photograms might reveal the conditions in the vicinity of the hole, indicating, perhaps, the amount

⁽Cf. R. E. Peterson, Transactions, American Society of Mechanical Engineers, Oct. Dec. 1933.)

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of plastic deformation from the superelastic stresses there, and testing the suitability of the X-ray method for stress concentration studies.

Results—Two complete series of photograms were taken, including the areas on the outer surface at the lateral edge of the hole, the longitudinal edge of the hole, a point ¼ inch along the specimen from the edge of the hole, and a point at the center of the cross section. These gave photograms at maximum, intermediate, and zero stress levels. The photograms differed very little, although those from the area at maximum stress appeared to be slightly more diffuse than the others.

Conclusions—Evidence presented in section (D) below indicated that the similarity in the spot patterns from different points on this specimen could not be ascribed to the composition of the material (its relatively high carbon content). The patterns therefore seemed to indicate an abnormally small amount of plastic flow of the material, or perhaps a deformation confined to an abnormally small fraction of the grains struck by the X-ray beam. Calculations were then made of the size of the area that had been subjected to stresses above the yield point. An elasticity formula for the stress around a hole in a flat plate is sufficiently accurate to be used, and gives the stress in the tangential direction at the lateral edge of the hole as

$$S_t = S \left[1 + 0.5 \frac{a^2}{r^2} + 1.5 \frac{a^4}{r^4} \right]$$

where S is the nominal stress, S_t is the tangential stress at the lateral edge of the hole (at points 90 degrees around the hole from the direction of the applied stress) and at a distance r from the center of the hole, and a is the radius of the hole. For the specimen studied here, the formula indicates that the stress drops 10 per cent in going only about 0.007 inch from the edge of the hole. The layer of metal stressed above the yield point, the layer in which the deformation might have been appreciable, had therefore been almost completely etched away in preparing for the X-ray study.

D. Spring Steel (0.85 Per Cent Carbon)

A further study of the sensitivity of the spot-pattern method was made on an annealed sample of high carbon spring steel which had been stressed in a rotating beam fatigue machine of the Moore

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type. The endurance limit of the annealed material was 34,500 pounds per square inch, the proportional limit 21,000, the yield point 36,500, the tensile strength 85,000, and the elongation 36 per cent. The stressing had been carried out three years prior to the X-raying.

After 18,000,000 cycles at a maximum stress of 33,500 pounds per square inch (1000 pounds per square inch below the endurance limit), the unbroken specimen was X-rayed at various places on the surface, and at another point where neither the bending stresses nor the static stresses from the grips were appreciable.

Results—Areas at the maximum stress, 33,500, gave photograms with more distorted spots than the area at zero stress, as will be seen in Figs. 9a and 9b. On the other hand, areas at stress levels of 28,000 and 20,000 gave diffraction patterns indistinguishable from the one with zero stress.

Conclusions—Stressing below the endurance limit causes plastic deformation, decreasing in amount with decreasing stress level, but clearly evident in photograms taken after 18,000,000 cycles 1000 pounds per square inch below the endurance limit. There seems to be no marked difference in this respect between 0.85 per cent and 0.19 per cent plain carbon steels (compare Fig. 9b and Fig. 6b).

E. Bessemer Screw Stock

A series of specimens that had been chosen as examples of a 0.20 per cent carbon steel were found to give photograms typical of cold-worked material, regardless of the place X-rayed. All spots were so diffuse that they overlapped to form a continuous ring. Inquiry then disclosed that the material was Bessemer screw stock in the cold drawn condition, which would preclude the detection of a small amount of additional plastic flow by spot patterns.

F. Annealed Copper

An extended investigation of copper by this method does not seem warranted. The stress-strain curve for annealed copper is curved even with low stresses, and stressing well below the endurance limit would therefore be expected to cause considerable deformation. Furthermore, studies on broken specimens would be meaningless since the specimens invariably receive a severe bend at the instant of fracture that would mask any deformation present just prior to the

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time of fracture. One specimen, however, was X-rayed. It was a Moore type rotating beam specimen of 0.324 inch minimum diameter that had been stressed the previous year. It had withstood, without breaking, 312,000,000 cycles at 9,900 pounds per square inch just below the endurance limit (about 10,000 pounds per square inch).

Results—Diffraction spots from stress levels under the endurance limit, at 9,900 and 6,900, were slightly diffuse as compared with spots from a point on the axis, under the grips, where the stress was negligible. The effect was not as distinct as with the steels, partly because of the larger grain size encountered here, but it was obtained on two different series of photograms and appears to be real.

G. Aluminum Alloy 25 S-T

Specimens of the duralumin type alloy 25 S-T were first chosen from among various aluminum alloys because of the uniformity and stability of the structure and because the S-N curve for this material is steep enough to provide a relatively wide range of stresses in a series of fractured specimens. (The analysis and history of the material is given in the footnote in the aluminum alloy portion of the section on widening of lines.) It was at once discovered, however, that the grains in the specimens were too large: normally only two or three grains reflected at a time and these could not be relied upon to represent the average condition in the specimen. The only conclusions of importance from the exposures on this alloy concern the surface of the break, where there was sufficient deformation to provide a much smaller effective grain size.

Results and conclusions—The fractured end of a specimen that had broken after 627,300 cycles of 20,000 pounds per square inch was X-rayed at several places. Photograms indicated that the grains at the surface of the smooth part of the fracture were less severely deformed than those on the rough part of the fracture; while there were badly blurred spots on the photogram of the smooth part, the spots from the rough area were so diffuse that they formed a continuous ring of almost uniform density. This would suggest that the deformation along the walls of a progressing fatigue crack is less severe than on the area of final rupture. On the basis of this observa-

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tion, Regler's claim¹⁹ that broken surfaces have a uniform line width characteristic of the material and independent of the type of break does not seem reasonable.

H. Alloys of Aluminum, 2 S-O and 3 S-O

To obtain a stable aluminum alloy of small and uniform grain size it seemed best to turn to the non-hardenable types of common alloys, as these had not been given a solution heat treatment. By

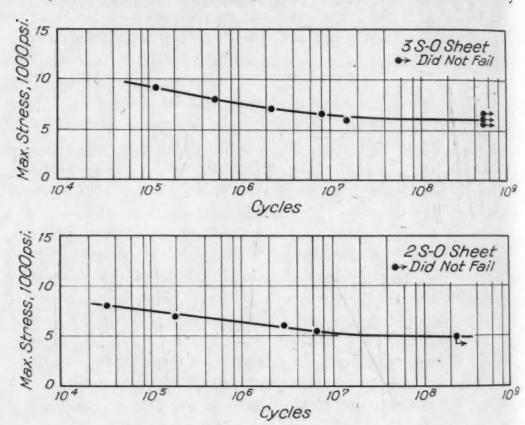


Fig. 10a and b-Fatigue Characteristics of 2 S-O and 3 S-O Sheets.

using both rotating beam and constant deflection sheet types many stressed specimens were available for study; S-N curves are given in Fig. 10.

Alloy 2 S-O is 99.2 per cent Al, containing Cu, Si, Fe as minor impurities, and 3 S-O is similar except for the addition of 1¹/₄ per cent Mn. The sheet specimens were stressed in 1933 and 1934, the

¹⁹F. Regler, Schweizer Bauzeitung, Vol. 105, No. 2, Jan. 12, 1935, and elsewhere.

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rotating beam specimen in 1928; the X-ray exposures were made in August 1935.

As the surfaces of the sheet specimens had not been cold-worked by machining and were in condition to give sharp spots, it was unnecessary to etch these.

Results—The results for these materials are summarized in Table II. In nearly all cases exposures were made at two different areas that had been subjected to the same stress, but in no case were there significant differences in the two patterns of a pair. A total of 23 exposures was made.

Conclusions—It will be noticed from Table II and the representative films reproduced in Fig. 11 that the patterns seem to fall into two classes, those with sharp or nearly sharp spots, as in Figs. 11a and 11b, and those in which plastic deformation has multiplied and blurred the spots until they form practically continuous rings, as in Figs. 11c and 11d. It will be further noticed that the former class invariably corresponds to stresses below the yield point, the latter to stresses above the yield point. On the other hand a similar distinction cannot be drawn for stresses above and below the endurance limit, for both a specimen stressed at the endurance limit and one stressed above the endurance limit gave sharp spots. It appears, therefore, that in these materials it is the relation of a stress to the yield point, and not to the endurance limit, that is the determining factor for the severity of the deformation and the type of X-ray pattern.

General Discussion of Spot Distortion—From the findings reported in this section the conclusion must be drawn that changes in diffraction patterns may occur during repeated stressing at stresses

Table II
X-ray Exposures of Common Alloys of Aluminum

Number of cycles	Stress at place X-rayed	Appearance of pattern	
0 435,000,000 505,000,000	0 2,170 2,680 3,000 2,840 3,550 4,000	Sharp spots Sharp spots Sharp spots Sharp spots Slightly diffuse spots Rings Rings	2SO rotating beam specimens: yield point 3,150; endurance limit 5,000
230,000,000 32,000	5,000 8,000	Sharp spots Rings	2SO sheet speciments: yield point 5,650; endurance limit 5,000
527,000,000 121.800	6,000 9,000	Sharp spots Sharp spots Rings	3SO sheet specimens: yield point 7,000; endurance limit 6,000

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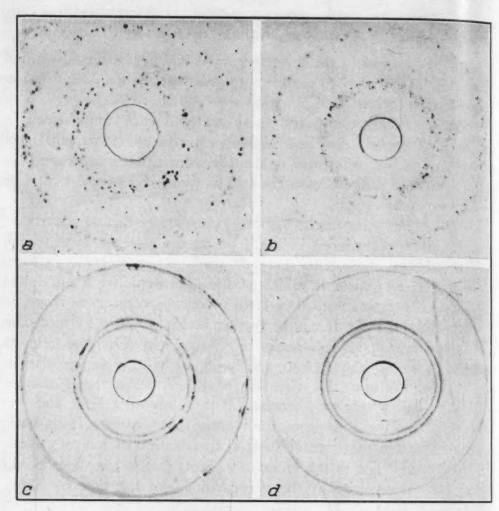


Fig. 11—Photograms of Aluminum Alloys 2SO and 3SO. (a) Unstressed. (b) Stressed 527,000,000 Cycles at the Endurance Limit and Below the Yield Point. (c) Stressed 505,000,000 Cycles Above the Yield Point and Below the Endurance Limit. (d) Stressed 32,000 Cycles Above the Yield Point and Endurance Limit.

within the safe range and, consequently, that they do not necessarily imply an impending fatigue break. This is in accord with the fact that slip lines are observed in the safe range of stress. The results further indicate that there is no abrupt increase in deformation (or in the resultant blurring of spot patterns) when the alternating stress increases from the safe into the unsafe range. In some materials deformation of the material is small when the stress is less than the yield point, but it appears probable that some slip occurs at all stresses above the proportional limit, and that the amount decreases asymptotically toward zero at still lower stresses. The present research is not primarily concerned with how the amount of slip varies with the number of cycles at a given stress level; at stresses below

the endurance limit it may occur near the start of the cycling and cease when the metal is sufficiently hardened by the stressing, as has been indicated by some observations on slip lines,²⁰ or it may be a steady progressive effect or possibly even an irregularly progressive one.

It does not follow, however, that X-rays are useless in fatigue work, simply because a single photogram of a shaft or propeller blade cannot reveal whether or not fatigue failure will occur. There still remains the possibility that the probable life of an object can be inferred from the appearance of a spot pattern, if the pattern is compared with a standard set of patterns made from fatigue specimens of the same material. The number of cycles of stress that the object had undergone would doubtless have to be known, at least approximately, for such a test, since a small number of cycles of intense stressing might give the same grain distortion as a larger number of cycles at a lower stress level; and, of course, there would always be a need for caution against being misled by any accidental deformation that might have occurred during service or variations from point to point in the material arising from its earlier history.

The simplest practical test procedure based on this principle would be to determine whether or not an object is being stressed so severely as to cause a change in the X-ray spot pattern after long continued stressing. If a change is found to occur, the active stress could be assumed to be above the minimum stress that is found to produce a change in spot patterns from fatigue specimens of the same material in the laboratory. The present experiments indicate that this minimum stress would not be the endurance limit, but a stress below this and related to it by a determinable factor which might be arbitrarily chosen as the factor of safety. It is not clear from the present data how many stress reversals would be necessary before such a test would be dependable; it can be stated, however, that most of the specimens showing changes below the endurance limit in the present study had received several million reversals, and the 0.19 per cent carbon studies indicate that the test would be unreliable with only a few hundred thousand reversals. It would be necessary in applying this test, of course, to protect from abrasion the small area on the highly stressed portion of the object which is to be used for X-ray examination unless it is possible to etch the spot deeply enough to remove all the effects of abrasion.

²⁰H. J. Gough, *Transactions*, American Society for Testing Materials, Vol. 33, Part II (1933) Edgar Marburg Lecture.

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IV. On the Quantitative Evaluation of Films

A number of attempts were made to find a simple way to determine the average amount of blurring or distortion of spots on a film. The ratio of the number of sharp spots to the number of fuzzy spots was first tried, ignoring the spots of uncertain sharpness. It was found that the readings of different observers did not agree—in fact they sometimes differed by a factor of two—and even that the same observer obtained different results for a film on different days. However, an observer reading a series of films at one sitting usually confirmed his classification of the films on the basis of general appearance, provided a sufficient number of spots were measured to give a good average.

The second method tried was that of counting the total number of abnormally elongated spots on the film. This proved valueless as it failed to distinguish slight degrees of blurring that could easily be seen in the general appearance.

The third method tried, one that has been highly recommended by Regler, was to measure the diameter of the spots in the direction of the periphery of the Debye rings, thus determining the average "peripheral width." Measurements were made directly from the films with a Brinell microscope, x10, omitting overlapping and underexposed spots. The total number of spots per film that could be measured was usually 30 or 40. Some of the results are summarized in Tables III and IV. With the 0.19 per cent carbon steel the results of three reading of films from the unstressed material were, respectively, 0.57, 0.72, 0.44 millimeters. With variations of this magnitude it was concluded that the labor required to make the

Table III
Peripheral Width of Spots from Annealed Copper*

	Depth Below		ripheral Width	
Stress Level Lbs./Sq.In.	Machined Surface Inch	First Reading	Second Reading	Appearance
0	0.015	0.54	0.58	Sharp
6900	0.015	0.54	0.66	Slightly blurred
9900	0.015	0.62	0.65	Slightly blurred
6900	0.003	1.50	1.50]	Progressively
6900	0.004	1.43	1.36	increasing
6900	0.005	1.38	0.84	in
6900	0.007		0.70	sharpness
6900	0.010	0.72	0.67	
6900	0.015	0.62	0.65	

*Cf. Section III, F

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Table IV
Peripheral Width of Spots from 0.19 Per Cent Carbon Steel*

Number of Stress Level		ripheral Width limeters	
Number of Stress Level Lbs./Sq.In.	First Reading	Second Reading	Appearance
300,000 at 25,000	0.75 0.76	0.77 0.83	Mostly sharp Slightly diffuse
300,000 at 25,000 } followed by 300,000 at 26,000	0.82	0.79	Slightly diffuse
300,000 at 26,000 J Same conditions, second film	0.74	0.69	Slightly diffuse

^{*}Cf. Section III, A, Second progressive experiment.

measurements was not justified. It should be mentioned that spots in a pattern differ greatly in width—frequently as much as a factor of five—so that a large number must be measured to insure a statistical distribution giving a good average. In the early stages of deformation probably only a few grains undergo slip; this method of reading the film averages the spots from these grains together with the spots from undistorted grains and thus tends to submerge the effect into the variation of spot size caused by variation in grain size. This inherent insensitivity is made worse by the necessity of neglecting badly distorted and underexposed spots. While the average width confirmed in many cases the visual classification as to sharpness, it appeared to offer no advantage over the latter in return for the considerable labor of measurement.

V. MEASUREMENT OF DEPTH OF COLD WORK IN FATIGUE SPECIMENS

Spot patterns of the type used in section III are useful for determining the depth to which a metallic specimen is cold-worked by an operation on the surface. Studies have been made with this method on the depth of cold work from various cuts on the milling machine and lathe²¹ and on the shaper.²² During the present series of studies the method was applied to two fatigue problems where it proved to be fully satisfactory. The X-ray procedure was to make a series of back reflection spot patterns of the surface after various thicknesses had been etched from its surface. In case the specimen had a diameter of several inches, this procedure could be replaced

²¹L. Thomassen and D. M. McCutcheon, *Mechanical Engineering*, Vol. 56, 1934, p. 155. ²²M. Renninger, *Metallwirtschaft*, Vol. 13, No. 50, 1934, p. 889.

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by one in which the cross section was polished, deeply etched and exposed to the X-rays at spots various distances from the periphery. With X-ray radiation from a cobalt target the penetration of the rays into the material is negligible. A series of patterns showing the increase in sharpness with increase in depth below a cold-worked surface is reproduced in Fig. 12.

(A) It is known that surface-rolling improves the fatigue strength of materials. This effect has been used to advantage in the surface-rolling of shafts whose fatigue strength has been lowered

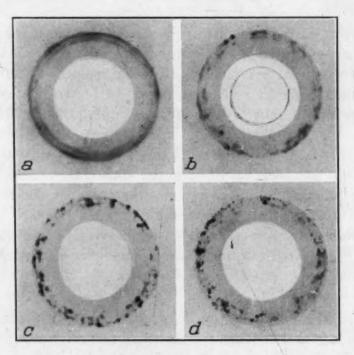


Fig. 12—Photograms Showing Depth of Cold Work in 0.19 Per Cent Carbon Steel. (a) Machined and Polished Surface. (b) Etched 0.0015 Inch Below the Polished Surface. (c) Etched 0.003 Inch Below the Polished Surface. (d) Unstrained.

by stress concentrations at the edge of a press-fitted wheel.²³ In this work it is desirable to know the depth to which the cold work extends when a shaft is given a certain rolling treatment. A problem of this type was attacked by the X-ray method with results that agreed satisfactorily with the results of other methods, as shown in Table V.*

²⁸R. E. Peterson and A. M. Wahl. *Journal*, Applied Mechanics (A.S.M.E.; Transactions) Vol. 2, March 1935 and discussions, June 1935.

^{*}The specimens were furnished by R. E. Peterson; the theoretical calculations were made by S. Way (Journal, Applied Mechanics, Vol. 2, June 1935); the Vickers hardness measurements were supplied by R. E. Peterson and A. M. Wahl, and the Rockwell readings by the author.

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Patterns from the first specimen listed revealed that all the grains were deformed to a depth of about 0.030 inch, that between 0.030 and 0.055 inch \pm 0.005 inch some grains were deformed and others were not, and that at greater depths there was no deformation.

Table V

Depth of Cold Work from Surface Rolling in 0.42 Per Cent Carbon Steel

Shaft Rolling Roller Contour				Depth of Cold-Worked Layer, Inches					
Shaft Diam., Inches	Rolling Load, Pounds	ad, Diam., Radius	Radius, Inches	X-Ray	Computed	Vickers Hardness	Rockwell "B" Hardness		
15/8 73/4	400 12,000	2 6	11/2	$0.055 \pm 0.005 \\ 0.44 \pm 0.03$	0.067 0.44	0.47 ± 0.05	0.50 ± 0.05		

It may be concluded that the X-ray method is reliable for such use and is applicable in some cases where other methods are not: in cases where the worked layer is too thin to permit hardness measurements or where hardness increase from cold work is too slight and microscopic evidence of plastic flow cannot be seen (as appears to be the case in medium and high carbon steels). It is, however, more time consuming than the hardness method.

(B) The endurance limit of steel is raised by cold work. Therefore a cold-worked surface layer of appreciable thickness could seriously alter the endurance characteristics of fatigue specimens—particularly with small specimens, where the cold-worked layer might amount to a considerable percentage of the specimen radius. These facts are commonly recognized in fatigue research and proper precautions are taken, but there seems to be no detailed study of the exact depth of deformation that is ordinarily encountered. This is important as a basis for estimating possible errors in the endurance limit and as a criterion for the proper depth to etch before undertaking X-ray studies of the type reported in sections II and III of this paper.

The results of a series of determinations on fatigue specimens of various materials are given in Table VI.

Another experiment in which the 0.19 per cent carbon steel was subjected to alternate polishing and etching showed, as expected, that polishing cold-worked the steel much less than the prior lathe work, the effect of the polishing extending to less than 0.0015 inch below the surface.

It will be seen that the R. R. Moore type fatigue specimens of

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Table VI
Depth of Cold Work in Fatigue Specimens

Material	Depth of Cold Work from Specimen Preparation, Inches
0.42 C Steel, hot-rolled* 0.19 C Steel, annealed* Annealed Copper* Annealed Aluminum (2S-O)**	$\begin{array}{c} \textbf{0.002} \pm \textbf{0.001} \\ \textbf{0.005} \pm \textbf{0.001} \\ \textbf{0.008} \pm \textbf{0.002} \\ \textbf{0.008} \pm \textbf{0.002} \\ \textbf{0.008} \pm \textbf{0.002} \end{array}$

*The machining procedure in preparing these specimens was to take a lathe cut of 0.015 inch followed by another of 0.015 inch, then by one of 0.002 to 0.003 inch, then filing and polishing to remove an additional 0.001 inch.

**The final operations were shallow lathe cuts, polishing with No. 218 Aloxite cloth, then with #00 metallographic paper, then buffing.

ordinary size when properly made are cold-worked to a depth not exceeding 1 to 5 per cent of their radius, and that the error in the endurance limit from this cause would not amount to more than 1 to 5 per cent (since the stresses at a point in the specimen are proportional to the radial distance of that point from the center.) A cold-worked layer of this thickness might be of considerable importance, however, in very small fatigue specimens, or in cases where stress concentrations are involved, for then the material at the maximum stress might lie almost completely within the cold-worked layer.

SUMMARY

Some 200 X-ray photograms of fatigue specimens of aluminum alloys, copper, and various steels show that, in general, changes in diffraction patterns of a material occur during stressing both above and below the endurance limit, and that only by comparing the patterns with those from specimens of the same material stressed under laboratory conditions could the extent of damage be estimated. A practical test based on the appearance of spot patterns ("asterism") is preferable to one based on the width of diffraction lines owing to the greater sensitivity of the former, in spite of formidable difficulties in making it quantitative. There appears to be no hope for an accelerated fatigue test involving X-ray patterns. X-ray diffraction is not a convenient method of investigating stress concentrations, as the volume of material at maximum stress is small and is frequently located in the cold-worked surface layers that must be removed by etching before X-raying. X-ray determinations of depth of cold work from machining agree satisfactorily with other methods which, though more convenient, are less generally applicable. The depth aration, Inches

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actually found in carefully prepared fatigue specimens varied from 0.002 to 0.008 inch with different materials.

ACKNOWLEDGMENTS

The need for research of this type was noted in 1934²⁴ and arrangements for the work were made by the A.S.T.M. Committee on X-rays; through the efforts of R. F. Mehl, Chairman, the cooperation of the A.S.T.M. Committee on Fatigue was secured and many valuable suggestions resulted, particularly from R. E. Peterson and R. L. Templin, throughout the course of the work since its initiation in March 1934. Preliminary reports on this work have been published in 1934, 1935, and 1936.²⁶

The author wishes to acknowledge the able assistance of G. Ansel, who did much of the work of etching specimens, making exposures, and reading films. It is also a pleasure to acknowledge the hearty co-operation of the staff of the research laboratories of the Aluminum Co. of America, the Westinghouse Electric and Manufacturing Co., and the National Tube Co., in providing materials and fatigue equipment.

DISCUSSION

Written Discussion: By N. P. Goss, metallurgist, Cold Metal Process Co., Youngstown, Ohio.

The author has shown conclusively that the X-ray method cannot be used in place of the accelerated fatigue test, and this is perhaps to be expected in view of the nature of the problem.

As I understand it, fatigue failure appears to be due to the spread of minute fractures, the spreading continues until the effective cross section of the test piece is reduced so much that it cannot support the working load on the test piece, so that failure ultimately occurs. The X-ray method of diffraction cannot show this.

Prof. H. F. Moore, who has studied this problem exhaustively and with great care, has defined the "fatigue limit" or the "endurance limit" of a test

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²⁴C, S. Barrett, Metals and Alloys, Vol. 5, 1934, particularly pages 158, 170, 173.

^{*}Semi-annual Progress Reports of the Metals Research Laboratory, Carnegie Institute of Technology, Pittsburgh, Pa., Oct. 1, 1934; April 1, 1935; Oct. 1, 1935; April 1, 1936. C. S. Barrett, Physical Review, Vol. 47, 1935, p. 333, (abstract), and Vol. 47, 1935, p. 338, (abstract).

C. S. Barrett, Journal, Applied Mechanics, Vol. 2, A-69, June 1935 (discussion).
Certain data were presented by Peterson and Wahl, Journal, Applied Mechanics, June 1935 and March 1936.

American Society for Testing Materials Committee reports covering the work were published in 1935 and 1936.

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piece as the lowest statistical unit stress at which failure will take place under repeated stress. The working stress is well under the ultimate static strength of the test piece.

In many instances, photomicrographs made of heat treated specimens stressed below the "endurance limit" show the familiar "slip bands," first observed by Ewing and Rosenhain. Failure cannot be attributed to the plastic flow, but due to the minute cracks or fissures.

It should be pointed out that the plastic flow, which may occur during fatigue testing and below the endurance limit, strengthens the test piece, but this is counteracted by the creation of fissures, and it is the cracks which cause the failure and unfortunately the X-ray cannot show this directly or indirectly.

The X-ray method can be used successfully to determine whether or not plastic deformation has occurred below the endurance limit, and the author has shown this very nicely.

On the other hand, the X-ray method fails when a test specimen in the cold-worked state is used, since it depends upon the changes in the "spot pattern." The author did not say anything definite about this, but it is implied in several portions of the paper.

I am also interested in knowing how the phrase "grain distortion" is interpreted by the author—(on page 1123) of his paper, to quote: "When the grains are undistorted, the atomic planes produce sharp spots while bending or distortion of the grains will lead to elongated spots and diffuse spots."

Asterism and line broadening, etc., have been oftentimes interpreted to indicate lattice distortion. However, it becomes apparent that some European investigators share the same viewpoint expressed by C. Nusbaum and myself, that asterism does not indicate lattice distortion, but gives evidence of "block displacement."

Many interpret asterism to mean lattice distortion, but a careful analysis shows this to be impossible. A recent paper by M. Kornfeld on "The Structure of a Deformed Crystal and Recovery Phenomena" supports the mechanism of plastic flow as one of block displacement.

Another question I would like to ask the author in regard to failure by fatigue: does it occur on the slip planes or on the cleavage planes?

It is usually believed by many that when a metal has been plastically deformed by cold working, the K^{α} doublets are not resolved; this is very important and since it is of importance in many industrial applications, I made a very careful investigation of this problem of K^{α} doublet resolution. The author states that (page 1120) when the surface is slightly deformed during the machining of the test piece, X-ray diagrams show that the K^{α} doublets are not resolved.

In my study of this problem, I used strip steels given area reductions in excess of 90 per cent. I found that the Ka doublets were always resolved provided the slit systems were properly constructed.

The Ka doublets were resolved when the following slit system was used: 9 centimeters long, and 0.015 inch aperture ¼ inch wide, using the same specimen and in exactly the same position, but in using slits having an aperture of 0.040 inch x ¼ inch wide, the lines were broad and the Ka doublets were not resolved. The X-ray beam was reflected from the surface of the specimen.

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A remark is necessary about the structure of the resolved doublets in these severely cold reduced strips; they lack intensity (reduced) and the Ka doublet lines are slightly diffused, though clearly resolved.

Tempering below the recrystallization temperature caused interesting changes in the sharpness and intensity.

The pinhole system used by the author (see page 1120) is too short and the pinholes too large for sharp line spectra. I am sure that an improved slit system having smaller pinholes and substantially longer would result in greatly improved X-ray diagrams.

It is regretted that after all these years, we have not adopted a standardized pinhole system, X-ray tubes and technique in general. Everyone uses a different size pinhole system and X-ray tube (in which the focal spot varies) and so many of our results can never be comparative. I hope that a standardized technique will be adopted by convention after a careful analysis of the problems to be met in practice has been considered. The sooner the better.

Oral Discussion

G. L. CLARK: I wish to express my own deep appreciation of this beautiful piece of work by Dr. Barrett, which certainly should stand as a landmark.

May I suggest, in connection with some of the diffraction patterns just shown, an observation of our own which might apply here—for example, if the grain size in Dr. Barrett's specimens had been just a little bit larger, then spots appear not only on the rings but with increasing size between the diffraction rings. When a specimen characterized by such a pattern is subjected to increasing stresses then we may observe the spots between the rings move toward the rings corresponding to particular planes and finally merge together in these positions leaving the space between rings entirely free from diffraction interferences. This phenomenon might well be of considerable usefulness in these fatigue studies in addition to the smudging of individual spots on individual rings as fatigue proceeds. I have wondered if Dr. Barrett observed this in any of his work,

Another point of interest is whether it is possible to detect by the diffraction pattern an actual fatigue crack. In working with Prof. H. F. Moore and his associates on fatigue and creep of single lead crystals, we came across an interesting possibility. On a lantern slide I will show a pattern taken in the region of a crack produced in a lead specimen. The interferences are very distinctly feathered and irregular in shape. In another slide I will show the pattern from a large crystal of aluminum in a very nearly annealed state on which a scratch was made with a sharp instrument. Each spot now has an irregular outline instead of the usual symmetrical appearance. This variation is the same in each spot and is magnified as the distance of the spot from the parallel X-ray beam is increased. Reasoning by analogy, therefore, a discontinuity in the surface of the specimen will result in the formation of this particular type of diagram, which is obtained when a crack is produced across the face of the crystal specimen in the field of the X-ray beam. The feathering of the spot diagram is, therefore, the result of a change in orientation of

¹Professor of Physics, University of Illinois, Urbana, Ill.

a section of a crystal caused by a crack. The re-entrant feature of the outline of the spot is a magnification of the crack itself. It is possible that this interpretation of these results may allow the detection of minute cracks formed during the first stages of failure. Obviously, the method is limited to the range of grain size which will yield spots and not smooth Debye-Scherrer rings.



Single Grain of Armco Ingot Iron X-ray Beam Transmitted Perpendicular to Surface. Molybdenum Ka, K β and K γ and Continuous Radiation. Tube Operated at 35,000 Volts M.A. 20. Innermost Spots are Due Entirely to Continuous Radiation. Some of the Spots are Due Both to Continuous as Well as Characteristic Molybdenum Radiation. Specimens Furnished by Lawrence Ott, Western Reserve University.

N. P. Goss: Split or resolved diffraction spots can be obtained by X-raying a single crystal which has been deformed slightly. The X-ray diffraction spots will be radial in character, but the striations (referring to resolved structure of streak) will make various angles with the axis of the radial streak (asterism). This is well illustrated in the accompanying figure above. Here some of the streaks are split radially, as spot No. 1, but the majority are com-

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²Metallurgist, Cold Metal Process Co., Youngstown, Ohio.

of the outline tat this interracks formed mited to the ebye-Scherrer posed of striations making various angles with the radial axis of the asterism, examine spots Nos. 2, 3, 4 and 5.

I would like to ask Dr. Barrett, just how many X-ray patterns he made of each specimen, i.e., were diagrams (X-ray) made before and after stressing, as well as intermediate, and was the same spot of the specimen X-rayed each time. In my opinion it seems reasonable to conclude that unless the same point on the specimen is X-rayed during the course of the fatigue testing, then the X-ray patterns would not be comparative. Apparently most of the specimens were X-rayed only after fatigue testing. Is that correct?

The blurring of the spots could also be due to the cold working effect resulting from the machining of the specimens, (cold working would fragment or displace the blocks). Etching would remove the layers disturbed by machining providing it was deep enough. I would like to ask Dr. Barrett whether he made allowances for this?

Author's Closure

This paper is not concerned with the nature of the slip and cleavage mechanisms in crystals, to which Mr. Goss' first questions refer; recent books on the plasticity of crystals have summarized the state of knowledge in this field, and the author has recently published a review of the extensive researches on the effect of deformation and internal stresses on diffraction patterns. A further discussion is unnecessary here, for the interpretation of the spot patterns in the present paper does not hinge upon any theories or details of such mechanisms, but is concerned merely with the fact that reasonably perfect grains produce fairly sharp spots, and that after plastic deformation these grains (and other similar ones) will produce spots that are blurred and elongated compared with spots from the undeformed material.

It is obvious that the patterns from the deformed material must always be judged by comparison with patterns from undeformed material, and this was done throughout this research. Two methods are possible for the purpose.

(1) The spots from the same grains may be observed before and after stressing. Theoretically, this is the more desirable method, but it is difficult in the laboratory and in practical applications. (2) The average condition of the spots in the undeformed and deformed states may be compared. This method requires that the same average sharpness of the spots be found at different points on the specimen before stressing; and that after stressing those areas subjected to the same stress level give the same average condition. This is the method used in the present study. These precautions were constantly in mind, and by choosing suitable materials and preparing patterns from different areas as a check, assurance was obtained that these requirements were met. However, it is not implied that they are met in all materials.

As has been stated in the paper, care was taken to etch deep enough to remove surface layers that had been cold-worked by machining. For example, the depth of cold work in aluminum was about 0.008 inch (Table VI), and the machined surfaces of the aluminum specimens were etched 0.015 inch before X-raying. The test for this is simple: when further etching causes no further sharpening of spots, the cold-worked surface has been etched away.

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³C. S. Barrett. "Internal Stresses," Metals and Alloys, 1934, Vol. 5, p. 131, 154, 170, 196, 324.

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Reference to the paper will show that the following statement of Mr. Goss' discussion is incorrect: "The author states that (page 1116) when the surface is slightly deformed during the machining of the test piece, X-ray diagrams show that the Ka doublets are not resolved." Lack of doublet resolution was noted at the surface of the fatigue break, but the doublet in Fig. 12 will serve to indicate the condition in the polished layer of a mild steel fatigue specimen, and here the doublet is resolved. It is recognized by all observers, I believe, that the slit system, the radiation used, and the amount of cold work all govern the resolution of the Ka doublet."

With regard to the choice of the pinholes described on page 1120, which is criticized by Mr. Goss, it should be emphasized that for producing spot patterns such as Figs. 6 to 12, the pinholes should usually be larger and the beam more divergent than for producing Debye rings for the line width measurement that Mr. Goss has in mind. It is necessary in preparing spot patterns to have pinholes that will allow the X-ray beam to diverge and strike a large number of grains in the specimen, for only then will there be enough spots on the film to show the average condition of the grains. Increasing the size of pinholes does not have the detrimental effect in spot patterns that it has in Debye patterns. There is an upper limit to the useful pinhole size, however, owing to the fact that when it is too great there are too many spots on the pattern, and the spots overlap and cannot be seen as individuals.

The spots lying between the rings, which are referred to by Professor Clark, are not ordinarily seen in back reflection patterns as they are in forward reflection patterns. This results from the fact that they are Laue spots made by general radiation. In the case of a polycrystalline sample, there are so many reflections that they overlap to a great extent, and furthermore, each of these spots is very weak because of the low intensity of the general radiation from copper and cobalt targets.

The movement of spots discussed by Professor Clark is not shown well in the type of diffraction patterns used in the present study, but can be shown in patterns made with a camera in which the specimen and film are mounted on the same carriage and oscillated together through a small range of angles, with the same grains remaining in the beam throughout the oscillation. The spots are then made with the characteristic radiation only, and their appearance is not distorted by the distribution of intensity in the continuous spectrum and by the variation of efficiency of the photographic emulsion for the different wave-lengths in the spectrum, as it is in the Laue method. The application of this camera to polycrystalline grains distorted by static stressing and fatigue stressing is being discussed in another publication,4 but I should like to mention here that the patterns made in this way indicate that the lattice of a polycrystalline grain is progressively distorted into wider ranges of orientation when it is subjected to increasing elongation in tension or increasing stress amplitude in fatigue, and that in general this distortion is about several axes because of the complex stress conditions surrounding a grain in an aggregate when deformation is occurring.

^{*}C. S. Barrett, "The Distortion of Grains by Fatigue and Static Stressing," Metals and Alloys, 1937, (at press).

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CONVERSION OF TENSILE TEST DATA FROM ONE FORM OF TEST PIECE TO ANOTHER

The Relation of Uniform Elongation to Total Elongation and Reduction of Area

By E. J. JANITZKY

Abstract

This paper presents the derivation and use of a mathematical equation for the conversion of the elongation percentage obtained on a tensile specimen of any practical diameter and gage length to the percentage which would be obtained on a specimen of standard dimensions. This equation makes possible the standardization of data obtained from specimens, which cannot be made to standard dimensions, and also a direct comparison of the data obtained in foreign countries on their respective standard specimens.

The derivation of the equation is based on the consideration that the elongation percentage is the sum of the uniform elongation percentage and the elongation percentage which takes place during local constriction. The uniform elongation percentage is independent of gage length while the local constriction elongation percentage is shown to be proportional to the diameter: length ratio.

Although the value of the uniform elongation percentage is desirable, it is not always available. Therefore, an empirical equation is derived from experimental data, so that the uniform elongation percentage can be calculated from the total elongation and reduction of area percentages.

It has long been known that the results of a tensile test may vary when the size and shape of the tensile specimen itself is varied when examining identical material. This applies particularly to the percentage elongation. Therefore in order to have a single representative figure for any given material, it is necessary to standardize the size and shape of the tensile specimen. In this country the standard specimen has a 2-inch gage length and a diameter of 0.505 inch between the gage marks¹. This standardization permits the accumulating of empirical data on various steels, so that the data may be used for engineering purposes.

A paper presented before the Eighteenth Annual Convention of the Society held in Cleveland, October 19 to 23, 1936. The author, E. J. Janitzky is consulting metallurgical engineer, Carnegie-Illinois Steel Corp., Chicago. Manuscript received June 19, 1936.

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Data are, however, not always available on a standard test piece. As an example, it is sometimes necessary to investigate pieces that are too small or too thin to permit cutting a standard test piece. Again, it is often desirable to study the data obtained in other countries, and it happens that the standard gage lengths and diameters, as used in England, France and Germany, vary for each different country. It thus becomes desirable to be able to examine the data obtained on some other size specimen and calculate what those data would represent on a U. S. standard (2 inch x 0.505 inch) test piece. To cite the figures, the standard English specimen is 2 inches x 0.564 inch; the standard French specimen is 100 millimeters by 13.8 millimeters or 3.94 inches x 0.543 inch, and the German standard is 200 millimeters by 20 millimeters, or 7.87 inches x 0.787 inch.

As to a test piece to be cut from a material too small for the U. S. standard, it is indeed possible to make use of the law of similarity¹ and if the deviation from the 2 inch x 0.505 inch size is not too great, the results will be fairly comparable when the dimensions are properly proportioned. However, the necessity for a translation from one form of test piece to another arises not infrequently, and it is the purpose of the present paper to present a method for making the calculation.

The essential feature in the present study is the observation of the amount of "uniform elongation." Similar earlier attempts have been made by St. Gallik² and by A. Krisch and W. Kuntze³. It may be well at this point to define what is meant by uniform elongation. This can be done best perhaps by a consideration of a typical stress-strain diagram, see Fig. 1. Up to the yield point there is no permanent elongation. Between the yield point and the point of maximum stress (the highest point on the curve), it is well known that the specimen elongates virtually without losing its cylindrical shape,—it simply becomes a cylinder of greater length and correspondingly lesser diameter. This phenomenon is realized by a few investigators and studied by them and it accords with the facts as observed in the present investigation. After the point of maximum stress has been reached the specimen elongates further but around the prospective point of rupture the area decreases more rapidly, causing the well known local constriction. The uniform elongation may

¹National Metals Handbook, p. 71 and 72 (1933).

²Internationale Stahl und Eisen-Normen, Budapest, 1930.

⁵Berechnung der Bruchdehnung einschnuerender metallischer Werkstoffe fuer beliebig grosse Messlaengen, Archiv fuer das Eisenhuettenwesen, Nov. 1933, p. 305-309.

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then be defined as that elongation which takes place between the yield point and the point of maximum stress. Fig. 1. In testing, the point of maximum stress may be determined by observing the point beyond which no further increase in load is required to break the specimen. This point is indicated by a drop of the beam which is readily distinguishable from the momentary drop of the beam which takes place at the yield point.

The elongation of a test specimen is then composed of two parts,

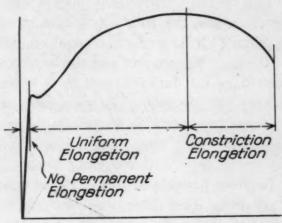


Fig. 1—Typical Stress-Strain Diagram Showing Uniform Elongation.

(1) the uniform elongation and (2) the additional elongation which takes place when local constriction occurs; thus, uniform elongation plus elongation during local constriction is equal to the total elongation. It becomes apparent that ultimate stress is the dividing index between uniform elongation and local constriction elongation. As to the uniform elongation, it will become apparent after simple consideration that the percentage uniform elongation is independent of the gage length. Uniform elongation is merely the result of change from one form of cylinder (with a certain diameter and length) to another cylinder which is smaller in diameter but longer. Therefore the percentage uniform elongation is independent of the gage length.

As to the constricted local elongation, which is the other term in the total elongation, this takes place only in a certain restricted region, not over the whole length of the test bar. The steel in this restricted region has already undergone uniform elongation and now (in this region alone) undergoes further local constriction elongation (and necessarily further reduction of area). The local constriction elongation is the elongation of this restricted region, and is thus a quantity

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to be added to the quantity of uniform elongation, the sum being the quantity of total elongation. By plotting experimental data it was observed that the percentage of local constriction elongation varies hyperbolically with the gage length. As the gage length increases in arithmetic progression, the per cent local constriction elongation decreases hyperbolically and becomes asymptotic to zero percentage local constriction elongation. Obviously the amount does not approach zero,—its percentage in relation to excessively long gage lengths approaches zero.

We find therefore that the total elongation in any specimen is composed of two parts, (1) the uniform elongation, whose percentage is independent of the gage length, and (2) the local constriction elongation, whose percentage can be expressed by a hyperbolic equation which becomes asymptotic to zero. The total percentage elongation being the sum of these two items, it becomes apparent that the percentage total elongation with very long gage lengths approaches asymptotically the percentage uniform elongation.

It therefore becomes possible to formulate an equation by which the percentage total elongation determined on one tensile specimen may be converted to the total elongation which would be obtained if the same steel were pulled in a tensile specimen of different gage length and/or diameter.

If we let

- T_1 = per cent elongation to be calculated on a new test piece.
- T = the experimentally determined per cent total elongation on a
- known test piece

 u = the experimentally determined per cent uniform elongation
- (this is independent of gage length)
 (T—u = the experimentally determined per cent local constriction elonga-
- tion the experimentally determined per cent local constriction elongation
 - L = gage length of specimen on which T and u are experimentally determined
 - D = diameter of gage length L
 - l = gage length specimen for which per cent total elongation is to be calculated
 - d = diameter of gage length of specimen for which total elongation is to be calculated
 - x = per cent local constriction elongation corresponding to gage length <math>l

Plotting of the experimental data has shown that the following proportion may be applied as to changes of local constriction elongation.

$$X:(T-u):\frac{d}{l}:\frac{D}{L}$$
 (1)

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(1)

That is local constriction elongation for any single material is proportional to the diameter: length ratio

The third and fourth term of the proportion (1) remain constant in a given transformation of one gage length to another, e.g. L d from the English to the French standard —— is constant, likewise

from the English to the German standard this ratio is also constant, but of different numerical value.

Thus we can rewrite proportion (1)

$$X = c (T - u)$$
where
$$c = \frac{L d}{l D}$$
(2)

As already mentioned the uniform elongation percentage is not affected by gage length and if added to equation (2) gives a general equation for conversion of one gage length to another, which may be expressed as follows:

$$T_1 = u + \frac{L d}{l D} (T - u)$$
 (3)

The ratio length between gage points to diameter in the

U. S. A. standard =
$$\frac{2 \text{ inches}}{0.505 \text{ inch}}$$
 = 3.96

English standard = $\frac{2 \text{ inches}}{0.564 \text{ inch}}$ = 3.456

French standard = $\frac{100 \text{ millimeters}}{13.8 \text{ millimeters}}$ = 7.246

German standard = $\frac{200 \text{ millimeters}}{20 \text{ millimeters}}$ = 10.00

Thus the conversion equation from English standard (T_e) to American standard (T_a) will read

$$T_a = u + (T_e - u) .895$$

and for the English standard to the French standard (Tt)

$$T_t = u + (T_e - u)$$
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Figs. 2 and 3 give a graphical comparison of observed total elongation and total elongation calculated by equation (3). Fig. 2 gives the total elongation for the U. S. A. standard test converted (by equation 3) from data experimentally determined on the English standard test piece. Fig. 2 also contains the total elongation for each

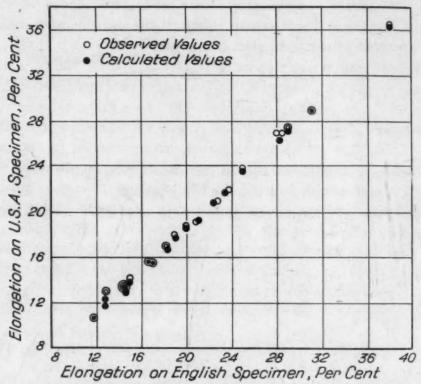


Fig. 2—Comparison of Observed and Calculated Total Elongations. English and United States Standards.

steel determined experimentally on English and U. S. A. standard test pieces. Black circles represent the experimental data, empty circles represent the values calculated by equation 3. Fig. 3 shows similar data converted from the English to the French standard test piece together with the total elongations experimentally determined in each of the two systems. Table I gives the total elongation and uniform elongation, determined on English standard test pieces, which were used in the conversion. The data in Figs. 2 and 3 and in Table I were taken from Jenkin.⁴ The uniform and total elongation for the English specimen were taken from Jenkin's table on page 7 of his book. All other data were taken from his Figs. 4 and 5.

⁴C. F. Jenkin-Report on the materials of construction used in aircraft and aircraft engines (1920) Figs. 4 and 5 tacing page 6 and table on page 7.

It will be seen that the above method of conversion from one

gage length to another demands a knowledge of the uniform elonga-

tion. Its amount may be observed easily while the tensile test is being

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Elongation on Trench Specimen, Per Central S

Fig. 3—Comparison of Observed and Calculated Total Elongations. English and French Standards.

Elongation on English Specimen, Per Cent

Table I

For U. S. A. and French Standard Test Pleces: A Comparison of Observed Per Cent Total Elongation and Per Cent Total Elongation Calculated from Tensile Data Determined on the English Standard Test Plece. Experimental Data were Obtained from Figs. 4 and 5 of C. F. Jenkin's Book⁴

Item	Ultimate Stress Tons/Sq. Inch	Meas	ngation ured on Test Piece Uniform	U.S.A.	longation Standard Calculated	Total Elo French S Observed	
1	68	20.0	7.0	18.8	18.6	13.0	13.4
2	61	22.5	8.0	21.0	20.9	15.0	15.1
3	46.1	28.4	13.0	27.0	26.8	19.5	20.5
4	59.1	23.5	8.0	22.0	21.8	15.3	15.6
5	79.8	16.7	6.0	15.5	15.6	11.1	11.2
6	65.7	21.1	4.5	19.2	19.3	12.5	12.6
7	38.2	29.0	11.0	27.5	27.1	_	19.8
8	37.9	31.1	11.1	29.0	29.0	21.0	20.8
9	40.0	25.0	12.0	23.8	23.6	18.0	18.3
10	37.8	29.0	11.0	27.5	27.1	19.8	19.8
11	. 66.2	19.0	8.5	18.0	17.8	13.5	13.6
12	84.4	12.8	3.5	13.0	11.8	7.9	8.0
13	63.2	17.0	3.5	15.5	. 15.5	10.0	10.1
14	82.0	14.8	5.0	14.0	13.7	9.9	9.8
15	46.8	28.3	11.0	27.0	26.4	20.5	18.4
16	28.0	38.0	22.0	36.5	36.3	28.0	29.8
17	110.6	11.8	2.0	10.8	10.7	6.8	6.8
18	102.0	12.8	3.0	13.0	12.4	7.2	7.8
19	94.0	14.5	3.5	13.5	13.3	8.8	8.8
20	87.0	14.5	4.0	13.5	13.4	9.0	9.1
21	68.6	18.2	5.0	17.0	16.8		11.4

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Table II
Comparison of Actual and Calculated Values for Uniform Elongation

1	2	3	- 4	5
Item	Total Elongation	Reduction of Area	Actual Uniform Elongation	Calculated Uniform Elongation
1	12.5	32.0	7.0	8.7
2	16.0	50.8	7.0	9.1
3	18.0	60.0	7.5	9.4
4	19.0	58.6	8.7	10.2
5	22.5	59.6	- 11.5	12.2
6	24.0	60.0	12.0	13.3
7	22.5	48.9	12.9	14.1
8	26.5	58.0	13.0	15.5
9	29.8	75.3	15.6	15.1
10	29.0	58.6	16.5	17.5
11	36.0	75.4	20.5	19.8
12	40.0	80.0	21.0	22.3
13	34.0	58.6	21.0	21.7
14	37.5	78.0	21.5	20.5
15	50.0	58.6	44.5	43.0
16	57.0	63.5	50.5	50.8
16 17	57.5	62.2	52.5	53.1
18	62.5	72.9	55.4	53.1
19	64.5	68.6	58.5	60.3
20	64.0	67.0	59.5	61.1

made, but on the other hand it is sometimes desirable to make such conversions from tensile tests which have already been carried out and in which the amount of uniform elongation was not recorded. Where it was not recorded, the only data available on the deformation are the total elongation and the reduction of area. It is therefore important to know whether the uniform elongation can be calculated from the total elongation and reduction of area. In order to secure data on this point, a series of tests was run on numerous grades of plain carbon and alloy steels, in conditions as-rolled, annealed and quenched and tempered. Austenitic steels were also included. The tensile tests were carried out on standard 2-inch x 0.505-inch test bars, and the uniform elongation, the total elongation and the reduction of area recorded. The deformation data so obtained are given in Table II.

In order to be able to obtain the uniform elongation when only the reduction of area and total elongation have been experimentally determined, it was necessary to obtain an equation relating these three properties. Such an equation was obtained empirically from the experimental data given in Table II. The general form of this equation is.

$$\sqrt[n]{\frac{R}{T}} = R$$

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or solving for u,

$$\sqrt[n]{\frac{\kappa}{T}}$$

$$u = \sqrt{R}$$
(5)

in which u and T have the same meaning as in equation (2), and R = the per cent reduction of area. As shown by columns 4 and 5 in Table II, this equation fits the experimental data over a wide range, namely 7 to 60 per cent uniform elongation.

In order to be able to obtain the value of the uniform elongation by substitution of experimentally determined values of total elongation and reduction of area in equation (4), it is necessary to obtain the numerical value of the root n. The root n of the deformation ratio (reduction of area to total elongation) obviously varies with gage length since the per cent total elongation increases with decreasing gage length and vice versa. The root n may be calculated for any gage length when total elongation, reduction of area and uniform elongation are given.

Thus solving equation (4) for n

$$n = \frac{\log R - \log T}{\log \left(\frac{\log R}{\log u}\right)}$$
 (6)

n = 2 for U. S. A. standard test piece, 2 inches x 0.505 inches gage.

Let us now take an example. Suppose we have a U. S. A. standard test piece on which the total elongation and reduction of area have been determined and for which we desire to calculate the uniform elongation so that we may then obtain the total elongation which the same steel would have if pulled in a German standard test piece. On the U. S. A. standard test piece the determined values were: total elongation = 34.6 per cent,

and reduction of area = 65.6 per cent.

The uniform elongation for the U.S. A. standard test piece is calculated from these data by sustitution in equation (5), thus

$$\log u = \frac{\log R}{\frac{1}{2} \log \left(\frac{R}{T}\right)} = \frac{\log 65.6}{\frac{65.6}{34.6}}$$

thus u = 20.6 per cent.

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By substitution in equation (3) we may then obtain the total elongation which this steel would have if pulled in the German standard test piece. Since the gage length of the German standard test piece is 200 millimeters and the diameter is 20 millimeters, and the gage length of the U. S. A. standard test piece is 2 inches and the diameter is 0.505 inch; the substitution in equation (3) will be:

$$T_1 = u + \frac{L d}{l D} (T - u) = 20.6 + \frac{2 \times 20}{200 \times .505} (34.6 - 20.6) = 26.1 \text{ per cent}$$

As a check, the total elongation was actually determined on a German standard test piece and the value 24.9 per cent was obtained.

It is also to be noted that only round specimens were considered and the material dealt with was steel.

Considering the coarse method of determining uniform elongation (by the drop of the beam at maximum stress) the data obtained by calculation do not show unreasonable spread.

Any mechanical improvement in instruments for recording the uniform elongation will be of benefit in the application of the conversion method proposed.

The writer also hopes to have made a contribution to the ever increasing inquiry into the meaning of uniform elongation and its application to steel engineering.

DISCUSSION

Written Discussion: By F. N. Menefee, professor of mechanical engineering, University of Michigan, Ann Arbor, Mich.

The writer has read this paper with great interest, and while he believes that there are limitations to the accuracy of the results that may be expected from computations based on T and u taken from one test specimen, and the practical effects of the small error in the basic assumption may set limits to the difference in size of test specimens between which the theory applies, the fundamental idea is good and the paper a contribution to the technique of interpreting test specimen data. There is a need for a means to transform data from one size test specimen to what that data would be if the specimen were different in size and the author can lay claim to having made an important step toward supplying it, in respect to the total per cent elongation.

In dealing with metals in the plastic zone the inaccuracies in our assumptions seem to magnify. Fortunately most of our assumptions hold fairly well within the elastic range and as a result our computations based on elastic theory are reasonably correct, although from actual tests made by the writer with a strain gage, it is his belief that many designers would be surprised if they actually knew how far the maximum stresses in their structures were from those indicated by their computations.

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fairly well astic theory riter with a sed if they were from In the case at hand the author has had to make assumptions, one of which is that a round test specimen will remain "virtually" cylindrical in shape during that period which he has called the period of "uniform elongation." Most engineers and metallurgists will recognize the practicability of this assumption knowing, of course, that it is not strictly the case and that there will be error in the conclusions drawn from it. Where that error does not cause the results to fall without the zone already set by other considerations the application of the author's theory will be very useful.

It is presumed that the author assumes the material to be uniform in structure, for to get a uniform percentage of plastic deformation between the yield point and the maximum strength, such a condition would be necessary, but it probably never is completely attained. For instance, in the three steels studied by the writer in connection with this discussion, the gage lengths of the German standard test specimens were subdivided into 10 short gages, and at the time of maximum load the elongation of each subgage was read as well as the total which was used to ascertain the author's value u.

From the data thus obtained it was found that the variation in per cent elongation between the subgages on the low carbon steel was from a low of 17.9 to a high of 24.6; on the medium carbon from 10 to 20.3 and on the high carbon from 11.5 to 16.6.

Table I Variation in Per Cent Elongation from Top to Bottom of Gage at Maximum Stress on German Standard Test Bars

Subgage Counting from Top .	Low Carbon	Intermediate Carbon	High Carbon	
1 2	17.9 21.5	10.* 18.	14.3 15.4	
3	21.5	19.2	16.6	
5	20.3 23.3	20.3 20.3	16.6 16.6	
6 7	24.6 23.3	15.9 17.3	16,6 16,6	
8	23.7	18.	15.4 15.4	
10	21.4 20.8	18. 12.8	11.5	

^{*}Influenced by proximity to shoulder.

It will be noted that the per cent elongation at the top and bottom are consistently lower than those near the center (due to influence of the shoulder) but that there are cases where there are variations near the middle of the gage which are due to lack of homogeneity of the steel.

In addition to the above, reference to Table IV will disclose another inconsistency which may be expected in the behavior of steel in the plastic range. Extreme care was taken to obtain the correct values of u for the various standard test specimens in low, medium and high carbon steel used by the writer; but there seemed to be little or no consistency, as shown by the upper part of the table, whereas the lower part shows variations in the total per cent elongations about as would be expected. The value of 9.4 for u on the low carbon French test piece was so far off that it was thought to be the result of an error until it was found that the elongations for the subgages totaled that recorded for the elongation of the whole gage length at maximum stress.

In passing, it is worth recording that there is a measurable difference

between the diameter at the center of a standard test specimen and the ends at time of maximum stress. For instance, on the English high carbon specimen, the reduction in diameter at the ends at maximum load was 0.022 inch, whereas at the center it was 0.045 inch. On the German high carbon specimen, the corresponding reductions were 0.028 and 0.051 inches.

The foregoing considerations are raised at this time to explain in part at least the departure of actual results from computed results in the writer's tests and to indicate why one may expect the theory to be limited in its accuracy on the common run of steels which have not been brought to a uniform physical condition.

The development of the theory while simple is ingenious and considering the limitations above set forth should give activate results.

To try out the theory developed by the author, the writer took three pieces of steel, two of which were 1-inch round and the other a deformed bar of concrete-reinforcing steel, 1-inch square, which fell into what is known as the "intermediate class" according to American Society for Testing Materials standards. It was the writer's idea to test the author's theory on low, medium, and fairly high carbon steel. Accordingly, of the other two pieces referred to above, one was low carbon and the other tool stock, although it had not been heat treated.

Each piece was large enough in diameter and length to get a standard American, English, French and German specimen from it, the length being something over three feet. The pieces were taken as they came from the stock piles, no attempt was made to bring each piece to a uniform physical condition throughout its length. It was recognized that it is possible for three feet of bar in a stock pile to possess different physical properties, due to unequal cooling conditions, but it was thought that many might want to know the limitations of the author's theory when applied to material in this way.

Each bar of stock was cut into four proper lengths to permit one American, one English, one French, and one German standard test specimen to be made therefrom. With slight variations, the specimens conformed to the dimensions referred to by the author on page 1150, such variations as \pm 0.002 inches in diameter were taken into account when computing areas and stress.

In running the tests from which the values of u and T were obtained in the accompanying tables, the yield point and maximum load were obtained as each test proceeded. The values are given in Tables II and III herewith to

	Yield Point	Table II in Pounds Per S	Square Inch	
Carbon	American	English	French	German
Low	36,500	36,200	34,100	34,000
Int. High	46,800 48,300	47,560 47,400	45,800 43,200	48,200 44,400
	Maximum Str	Table III	Square Inch	

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indicate what variations one may expect in these properties in one piece of steel a little over three feet long. The yield point was determined by the drop of the beam checked by dividers for measuring appreciable elongation on the test specimen.

Table V shows the results obtained. The relation between the per cent total elongation on the American and English test specimens bear about the same ratio as do the values given in the author's Table I. Likewise the accuracy of computed results as compared with actual tests is about the same. But in going from the American test specimen to the French and German there is a departure between the computed prediction and the actual test results. If this error is due to the computation being based on one determination of T and u on the American test specimen instead of the average of a number of tests, then Table V is faulty to that extent. It may be, however, that in going from the American test specimen to the German the disparity between sizes is so great that the errors in original assumptions are magnified and a correction factor will have to be introduced.

The writer believes the subject matter is worthy of more study than time permits preceding the reading of the paper. Out of its publication, however, there is bound to come an answer to the inaccuracy of the theory as applied to steels "as received" and possibly a factor to correct for it or specifications as to where it is applicable and where not.

Table IV Uniform Per Cent Elongation u

		Standard							
	Carbon	American 2" × .505"	English 2" × .564"	French 3.94" × .543"	German 7.87" × .787"				
	Low Int. High	17.5 19.5	20 15 15	9.4 11.7 15.5	21.38 17.3 14.63				
H.		Total	Per Cent Elor	ngation					
	Low Int. High	35.5 32. 30.5	37.5 33.5 32.	33.5 29.9 27.7	32.4 26.7 26.1				

Table V

Comparison of Actual Total Per Cent Elongation With the Computed Per Cent Elongation on English, French and German Standard Test Specimens, Based on Tests for Tandard u on an American Standard Test Specimen

			Per Cent Total Elongation English French				German—	
Carbon	Ame	rican u	Computed T ₁	Actual Test T	Computed T ₁	Actual Test T	Computed T ₁	Actual Test T
Low Int. High	35.5 32.0 30.5	19.0 17.5 19.5	37.4 33.62 31.8	37.5 33.5 32.0	28.06 25.38 25.54	33.5 29.9 27.7	25.54 23.24 23.86	32.4 26.7 26.1

Written Discussion: By R. L. Kenyon, research metallurgist, American Rolling Mill Co., Middletown, Ohio.

I have been interested in the matter of uniform elongation for some time, and discussed it somewhat at length in an article which I wrote for the October 1932 issue of *Metals and Alloys*.

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One of the questions that I should like to ask regarding the data in this paper is the method of determining the uniform elongation. As discussed in the article just referred to, this value can best be taken from an accurately drawn stress-strain curve. When the load on the specimen comes to its maximum value before fracture, two counteracting effects are taking place; a strengthening of the metal due to work hardening, and a weakening of the load-carrying capacity of the specimen due to reduction of area.

The cross section of the test piece is decreasing continuously throughout the test, but the work hardening factor predominates up to the point of maximum load. It is quite reasonable to assume, then, that there is a point where these two effects are equal although opposite. As a matter of fact, we generally find a flat place on the stress-strain curve at the maximum which looks like that shown in Fig. 8 of the article in *Metals and Alloys*. This is much more common than the type of curve represented in your Fig. 1.

When the load-carrying capacity of the specimen stops increasing, local reduction must have started at some point on the specimen. The end of the uniform elongation is, therefore, the point at which the load first reaches its maximum value. If the elongation is measured at the point where the beam drops on the old lever-type machine, this would be in error, due to the fact that the specimen had already begun to suffer local reduction.

In Professor Menefee's discussion of this paper, he quoted the results of an experiment in which he measured the elongation over different portions of the gage length just before the beam dropped. He found that greater elongation had taken place in the middle of the specimen than near the shoulders, and I believe that this agrees with the statements I have just made.

There is no information given in this paper as to the method of arriving at the uniform elongations given in Table I and II. If they have been determined by the drop of the beam, they are not really uniform elongation, and I should think there would be considerable question as to the validity of any equations derived from them.

All of the samples discussed in the paper are of a circular cross section, and the question at once arises as to the applicability of these formulas to flat specimens. The general relationships between the elongation values on specimens of different proportions, and of any shape, can be determined by reference to the slenderness ratio (gage length divided by the square root of the cross sectional area). It is much easier to prepare round specimens of definite proportions than plate or sheet samples, because the latter are customarily tested in whatever thickness they are rolled. For this reason, with a constant width and gage length, the slenderness ratio varies according to the thickness. The control of the slenderness ratio is much more difficult in this case than in the round specimens which are machined all over.

This entire subject is discussed at some length in the Metals and Alloys article.

Written Discussion: By R. L. Templin, Aluminum Company of America, New Kensington, Pa.

The author has discussed some fundamental conceptions of elongation of a test piece very much along the lines proposed first by Lebasteur and Marié¹

^{1&}quot;Commission des methodes d'essai des materiaux de construction." Official Report, Vol. I, page 120.

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gation of ad Marié¹ al Report, for round specimens and by Barba² for rectangular specimens. In spite of the fact that Martens in his classical Handbook of Testing Materials,³ points out that "the so-called proportional elongation no longer appears distinctly separated from the local extensibility near the gorge" when using short test specimens, Bartella elaborated on and developed the idea in his work on "Tension Testing of Materials." Bartella gave a new equation showing the relationship between

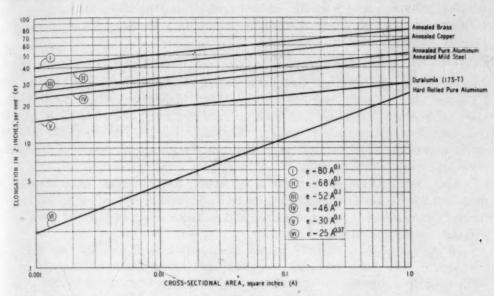


Fig. 1

elongation, gage length and material which was referred to and supplemented by additional data in a paper by the writer before the American Society for Testing Materials.⁵ While this last named work was based primarily on the results of tests of aluminum alloys, yet it was found to apply equally well to brass and mild steel results obtained by other investigators. In the writer's experiments it was found that cold working appreciably changed the slope of the straight lines obtained on the log-log plot as shown in Fig. 1. In this figure the abscissae are the cross-sectional areas and the ordinates are the elongations for 2-inch gage lengths. It was pointed out that for practically all types of standard test specimens, the relationships indicated are found to be applicable. More recently this work has been further amplified by Oliver.⁶

The author has adopted the well-known method of representing the elongation of his specimens as being made up of two parts and, for the uniform elongation over the gage length of the specimen, arbitrarily writes his Equation (4) which appears to be entirely empirical and extremely complicated. In Equations (4) to (6), the reduction in area at the point of fracture is the controlling factor in determining the uniform elongation distributed throughout the speci-

^{2&}quot;Resistance des materiaux." Mem. Soc. Ing. Civ. 1880, S. 682.

³Part I, page 122, Paragraph 143.

^{&#}x27;Italian Journal of Civil Engineers in 1922.

^{6&}quot;Effects of Size and Shape of Test Specimen on the Tensile Properties of Sheet Metals," Proceedings, A.S.T.M. 1926, Vol. 26, Part II, p. 378.

^{6&}quot;Proposed New Criteria of Ductility from a New Law Connecting the Percentage Elongation with Size of Test-Piece," by D. A. Oliver, *Proceedings*, Institution Mechanical Engineers (London), No. 4, December 1928.

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There is one limitation to these equations which has not been emphasized by the author and that is the fact that the reduction of area usually cannot be determined reliably on any but round specimens. Tests of round specimens of most structural materials represent only a relatively small part of the total number of specimens actually encountered. Consequently, the practical significance of these complicated formulas is not very clear.

Author's Closure

In regard to Professor Menefee's remarks about uniform elongation I wish to state that already Martens followed the same reasoning about uniform elongation when Tetmeyer proposed to use uniform elongation as a criterion for ductility. Nevertheless, Galick, Kurtze, Kirsch, and other collaborators on this question avail themselves of uniform elongation as means of translation from one gage length to another.

The introduction of a correction factor for different gage lengths as proposed by Professor Menefee, if he is right, would, according to his experimental data require a factor which would increase exponentially as the product of gage length and diameter increases.

In answer to Mr. R. L. Kenyon's comments I wish to state that Fig. 1 in my article was only intended as schematic representation. Regarding the method of determining uniform elongation, this was determined by the drop of the beam method by both Jenkins (whose data I used) and also by myself. I realize that this method is crude, and mentioned this on page 1158, lines 11 to 16, in my article. Undoubtedly an accurately determined stress-strain diagram is to be preferred. However, irrespective of the experimental error in any given method of determining uniform elongation, you will admit that as long as the elongation is uniform, the per cent elongation is independent of the gage length, and that constriction elongation is in inverse ratio to the gage length. That is, if total elongation is plotted against gage length for test pieces of the same diameter, a hyperbolic curve results which however does not descend to the abscissa but becomes asymtotic to the abscissa at the value of the uniform elongation for the particular steel being tested.

However, I cannot see that the inaccuracy of the experimental data resulting from the method of determining uniform elongation invalidates my conceptions, although it may be expected to cause a greater deviation in the results for any individual calculation. In order to be independent of the measurement of the uniform elongation, I developed equation 4 in my article; i.e., a relation between total elongation, uniform elongation and reduction of area. If there should be a small constant error in equation 4, because the drop of the beam method was used in determining the uniform elongation for developing this equation, I believe that the error could be corrected by adjusting the root n when more precise data are available.

As to the applicability of the conception to flat products, I limited myself in my paper to the discussion of round test pieces, but I believe that the method may be applied to flat sections by replacing the diameter in my equation by another conception.

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ited myself the method equation by In reply to the remarks of Mr. R. L. Templin, I wish to state that Bartella's equation disregards uniform elongation and assumes hyperbolic change of total elongation with changing gage length.

In my equation the total elongation has been considered to be made up of two parts, the uniform elongation and the elongation during local constriction in accordance with other investigators.

Of these component parts of the total elongation, only the constriction elongation is assumed to change hyperbolically with the gage length, the uniform elongation being independent. For this reason Bartella's equation approximates only within the range of gage lengths in which the constriction elongation forms a great part of the total elongation, but will not satisfy austenitic steels or steels with high uniform elongation. Thus it is apparent that different grades of steel would require different material constants, and the universality of Bartella's equation for steels is questionable.

THE ELECTRODE SALT BATH FOR HARDENING HIGH SPEED STEEL

By AXEL HULTGREN

Abstract

Based on twenty years' experience with electrically heated salt baths, the author describes the factors that cause decarburization, oxidation, and burning, of high speed steel tools. Data are presented on the use of silica in the salt bath to prevent decarburization. Photomicrographs showing the results to be expected in treating 18-4-1 and molybdenum-tungsten type steels illustrate the difference in results obtained with, and without, the use of silica in a bath of barium chloride. Immersed electrode furnaces are best for hardening high speed steels, but if improperly designed, may cause heating current to pass through tools, resulting in local overheating, and partial melting. The furnace, using closely spaced pairs of electrodes, overcomes the danger of local overheating.

THE salt bath for heat treating high speed steel has not been particularly favored in the U.S.A. This is remarkable in view of the fact that internally heated electrode salt bath furnaces have been used for that purpose for more than 30 years in Germany and Sweden and are today in fairly general use in those countries. Perhaps, therefore, some observations made and results obtained in this practice by the author will be of interest to American metallurgists.

The main qualitative requirements of a hardening furnace for high speed steel are, of course: uniform temperature, accurate temperature control and reasonable freedom from reaction between the surrounding medium and the steel surface, such as: carburization, decarburization or oxidation. If properly designed and maintained the electrode salt bath fulfills those requirements. Without certain precautions, however, serious difficulties may be encountered in the use of that furnace: decarburization usually accompanied by some oxidation and local overheating. In one respect, the salt bath is superior to other hardening furnaces: the salt film adhering to the work

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A paper presented before the Nineteenth Annual Convention of the American Society for Metals, Atlantic City, October 18-22, 1937. The author, Axel Hultgren, is Lecturer in Metallography at Tekniska Högskolan, Stockholm, Sweden. Manuscript received September 28, 1937.

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protects the latter from oxidation during the passage from the furnace to the quenching bath.

In principle, the electrode salt bath furnace consists of a thermally insulated refractory container for the salt, sometimes having a lining or crucible of steel, into which are introduced two or more electrodes of steel, alternating current of low voltage being passed between the latter through the salt. The salt may be BaCl₂, with or without additions, or a proprietary mixture. The temperature may be measured by the use of a thermocouple, an optical or a radiation

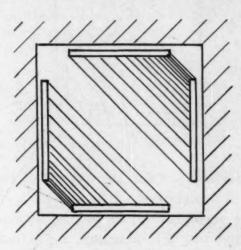


Fig. 1-Electrode Salt Bath.

pyrometer. In the last two cases, the instrument may be focussed on the surface of the salt—a more or less crude method, since it is difficult to prevent the salt fumes from interfering—or onto the bottom of a closed tube inserted for a sufficient depth into the salt and reaching out of the region of the fumes, thus insuring practically blackbody conditions. The temperature may be controlled automatically in different ways. It is not proposed to deal here with details of design except in so far as they are concerned in the observations made.

In 1915 to 1925, when associated with the SKF Company of Gothenburg, the author had occasion to work with a furnace of the type shown in Fig. 1. The crucible was lined with chamotte, the salt was unmixed BaCl₂ or, sometimes, BaCl₂ mixed with KCl. It turned out that this salt was decarburizing to high speed steel. For instance, 5 minutes immersion at 1310 degrees Cent. (2390 degrees Fahr.) would cause the formation of a ferritic skin 0.03 millimeter (0.0012 inch) thick or more.

Experiments made in 1916 to 1917 on a laboratory scale with

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crucibles made from magnesia, chamotte and silica indicated that only silica had the property of counteracting or preventing decarburization from the salt contained in the crucible. From that time on, silica bricks were used for lining the crucible of the furnace and the decarburization practically disappeared. The theory was that silica by combination removed from solution basic oxides that were responsible for the decarburization. The oxides might be derived

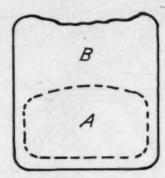


Fig. 2-Section Through Salt Ingot.

from reaction between the salt and the oxygen of the air or be introduced in the form of scale.

Later on, tests were continued by the author's successor, B. Kjerrman, who found that the decarburizing action of the salt was chiefly due to the presence of BaO and who developed the method of intermittent additions of ferrosilicon for counteracting decarburization.² At the same time, he substituted chamotte for silica as lining material, thus improving the life of the lining.

In the years 1926 to 1932, when at the Söderfors steel works, the author made various experiments with a similar salt bath furnace to that shown in Fig. 1. Without going into detail, it may be stated that the beneficial action of silica present in the bath was confirmed. The lining was made from chamotte and the silica could be introduced in various ways, for instance, by keeping silica slabs inserted in the bath. When a freshly lined crucible was filled with new salt it would take 3 to 4 hours at 1300 degrees Cent. (2370 degrees Fahr.) to complete the reaction with the silica and bring the salt into the nondecarburizing state. The continual reaction with silica causes the formation of a sludge that settles onto the bottom of the crucible from where it is removed from time to time with a scoop. However, this automatic purification of the salt may be more or less

¹U.S. patent number 1,713,472.

²Transactions, American Society for Steel Treating, Vol. 16, 1929, p. 392.

perfect depending on conditions, particularly on the temperature at which the sludge is removed. This is illustrated by the following data.

In 1929, after a long run of the furnace at 1300 degrees Cent. (2370 degrees Fahr.) using silica addition, the whole mass of salt was scooped out while at the high temperature and poured into a carburizing box, excluding the sludge. When the frozen salt ingot was broken up it was found to have segregated in two different portions as shown in Fig. 2. The lower central portion, A, was dark grey, the upper portion, B, had a light greenish appearance. On analysis the following results were obtained.

	A	В
Portion	Per Cent	Per Cent
Soluble in water	. 27.4	98.1
Insoluble in water	. 72.6	1.9
BaCl ₂	. 25.7	
BaO	. 30.1	
FeO	. 19.4	
Fe ₂ O ₃	. 4.4	
CaO	. 0.6	
Al ₂ O ₃	. 1.0	
SiO ₂	. 19.0	
Total	100.2	

It may thus be concluded that, very much like the settling of so-called free crystals toward the lower central portion of a large steel ingot during freezing, the impurities of the salt, which were soluble at 1300 degrees Cent. (2370 degrees Fahr.), during cooling were the first to separate and, owing to their greater density, settled toward the bottom, leaving the purer, more fusible salt at the top. The portion next to the wall froze more quickly and had insufficient time for separation. Obviously then, to keep the salt in good condition, the sludge should not be removed from the crucible until the salt has cooled from the operating temperature to about 1000 degrees Cent. (1830 degrees Fahr.).

In later years, firms making heat treating salts, in Germany and the U.S.A., have put on the market proprietary salt mixtures which, judging from the limited experience the author has had with one of them, give adequate protection from decarburization at high speed steel hardening temperatures.

For a routine decarburization test, the author has been using a small, smoothly machined rod of 18-4-1 steel, 8 millimeters round by 50 millimeters ($\frac{5}{16}$ by 2 inches). This is immersed in the salt for 5

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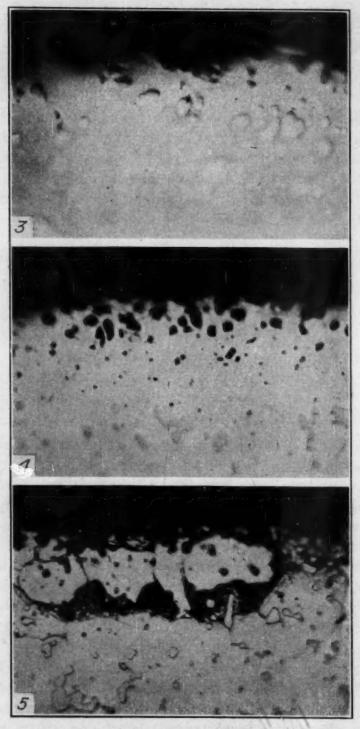


Fig. 3—High Speed Steel 18-4-1, O.-H. After 5 Minutes at 1310 Degrees Cent. (2370 Degrees Fahr.), Silica Addition. Unetched. × 1500.

Fig. 4—High Speed Steel 18-4-1, O.-H. After 5 Minutes at 1310 Degrees Cent. (2370 Degrees Fahr.), No Silica. Unetched. × 1500.

Fig. 5—The Same as Fig. 4. Etched. × 1500.

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minutes at 1310 degrees Cent. (2390 degrees Fahr.), quenched in oil, sectioned and examined in the microscope. Some results obtained in this way under various conditions are given below.

Fig. 3 shows the edge somewhat rounded from polishing—of a rod section, unetched, after treating in this manner, the salt being practically nonreactive owing to silica addition to the salt which was BaCl₂. There is hardly any oxide visible, nor was any ferrite seen after etching. Figs. 4 and 5 show a specimen, unetched and etched respectively, which was heated in the same way, after the silica addition had been omitted for some time. Oxide particles have formed near the surface to a depth of 0.016 millimeter (0.0006 inch), and there is a ferritic skin about 0.010 millimeter (0.0004 inch) thick. Another specimen was heated for 80 minutes, still without silica. Fig. 6 shows the unetched surface portion. Oxide particles are present to a depth of about 0.12 millimeter (0.005 inch), larger, crystallized opaque ones near the edge, small transparent globules further in. After etching, Fig. 7, the following structure zones are revealed in succession from the surface inwards:

The innermost transition zone is further shown in Fig. 8. The austenite is, of course, largely transformed to martensite but on the border lines between martensite and ferrite dark-etching areas of sorbitic or troostitic appearance are seen. The ferrite contains small particles, probably carbide particles precipitated during cooling.

The formation of this sequence of structure zones may be explained by reference to Fig. 9, which is really a section through the Fe-W-C diagram at 20 per cent tungsten according to Takeda,³ slightly modified, but may also serve to outline the phase regions traversed when high speed steel is decarburized. Assuming the steel in question at 1310 degrees Cent. (2390 degrees Fahr.) to be represented by point (1), the structure will consist of austenite and double carbide (η) . After a certain amount of carbon has been removed, to point (2), ferrite will also appear. On decarburizing further, as in point (3), austenite has disappeared and the structure consists of ferrite and carbide (η) and, finally, only ferrite will remain, as in point (4) of the diagram. This agrees well with the

The Technology Reports of the Tohoku Imperial University, Vol. X, 1931, p. 74.

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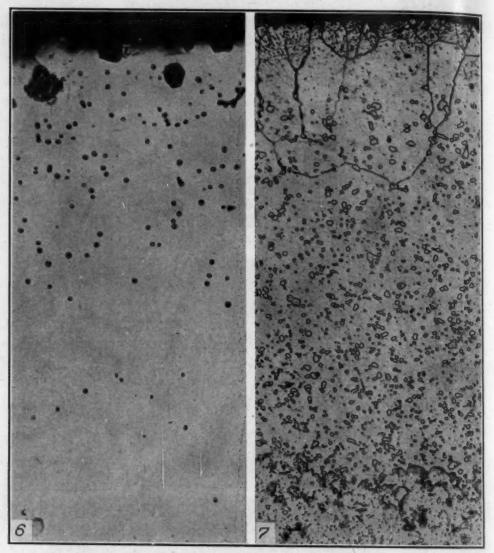


Fig. 6—High Speed Steel 18-4-1, O.-H. After 80 Minutes at 1310 Degrees Cent. (2370 Degrees Fahr.), No Silica. Unetched. × 750.

Fig. 7—The Same as Fig. 6. Etched. × 150.

structures found in Fig. 7. It is interesting to note that the ferrite shows a tendency to form columnar grains. This phenomenon has been dealt with elsewhere and attributed to progressive transformation caused by diffusion.⁴ In the field shown in Fig. 7 one of the columnar grains has outgrown its neighbors and occupies the main part of the field, an exaggerated case of "columnarity."

The sorbitic areas shown in Fig. 8 are believed to have formed in the following manner. Where austenite and ferrite co-exist, there will be an adjustment of the composition of those two phases

⁴Transactions, American Institute of Mining and Metallurgical Engineers, Institute of Metals Division, 1929, p. 594.

in the direction that ferrite will preferentially dissolve silicon, chromium and tungsten, austenite preferentially C and Mn. In quenching, during the first stage of cooling, equilibrium conditions will be altered, as seen in Fig. 9, in such a way that the proportion austenite to ferrite will increase, in other words austenite will invade the adjoining ferrite to some extent. It is not to be expected that

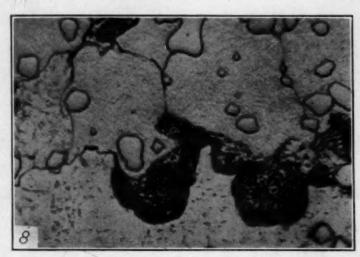


Fig. 8—The Same as Fig. 7. Transition Zone With "Sorbitic" Areas. Etched. × 1500.

those rapidly formed austenitic portions will have time, during quenching, to equalize their composition with the bulk of the austenite. In consequence, their alloy content, inherited from the ferrite, will not be suitable for hardening in oil, and they will decompose, during a later stage of cooling, into some kind of sorbitic structure. The marked, convex border-lines of those areas toward the ferrite support this explanation. Incidentally, such dark areas are often seen after partial melting of high speed steel. Sometimes, they have been mistaken for eutectic.

Reverting to oxidation, Figs. 4 and 6 demonstrated that oxide particles may form to some depth when high speed steel is heated in salt that is not free from basic oxides. The oxygen entering the steel will naturally first attack silicon, forming the transparent globules of silicate shown. Next chromium, (vanadium, tungsten and iron) will probably combine to form the opaque crystals nearer to the surface (Fig. 6). A continuous scale formation has not been observed after heating in salt.

The data given above on decarburization and oxidation of high speed steel 18-4-1 apply equally well to high speed steels of

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other compositions common in Sweden such as, for instance, steels of high cobalt content. In regard to steels of high molybdenum content, this ought to be true also, for the following reasons. If a high speed steel with high molybdenum is heated for hardening under conditions sufficiently oxidizing to cause the formation of scale the molybdenum oxide in the scale will volatilize, thus upsetting the

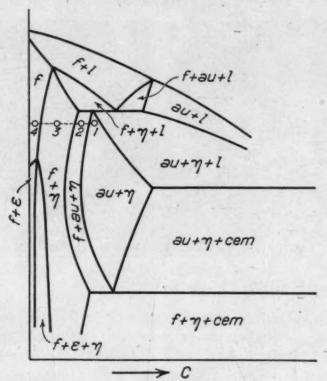


Fig. 9—Section Through Tentative Fe-W-C Diagram at 20 Per Cent Tungsten According to Takeda, Slightly Modified.

Note: f = ferrite, au = austenite, 1 = liquid, $\epsilon = tungstide$ (Fe₇W₆), $\eta = double$ carbide /(Fe, W)₆C₃/.

equilibrium between oxide and metal. In consequence, more molybdenum from the metal surface will react with the scale to form oxide and volatilize. In this way molybdenum will be pumped out of the steel surface, so to speak, and the surface portion, after hardening, will not have the alloy content required to give the desired hardness and other high speed steel characteristics. In salt heating under the controlled conditions defined above, it is not likely that the oxygen supply to the steel surface will be sufficient to oxidize molybdenum in the steel.

The principle of heating the salt internally presents obvious advantages but may, under unsuitable conditions, introduce a serious

ybdenum ns. If a ening unof scale etting the difficulty. The current, which is supposed to pass through the salt, takes, of course, the path of least resistance. If, in a furnace according to Fig. 1, a large object, say a milling cutter, is immersed in the salt the path of least resistance will lead through the steel as shown in Fig. 10. Thus the current density will rise abnormally,

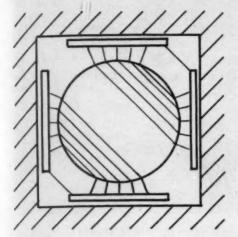


Fig. 10-Electrode Salt Bath with Large Milling Cutter Immersed.

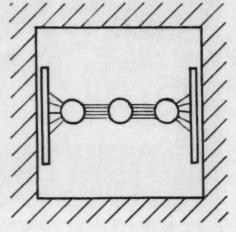


Fig. 11-Electrode Salt Bath with Taps Immersed.

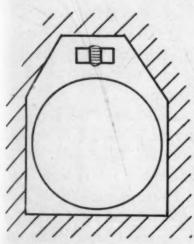


Fig. 12-Electrode Salt Bath, New Design.

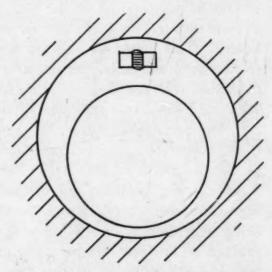


Fig. 13-Electrode Salt Bath, New Design.

near those portions of the tool that are turned toward the electrodes. Overheating and partial melting will be the result, without any indication from the pyrometer that anything is wrong. Similarly, three taps arranged in a row as shown in Fig. 11 showed signs of partial melting on the teeth coinciding with the plane through their axes.

To overcome this difficulty, the crucible must be made corres-

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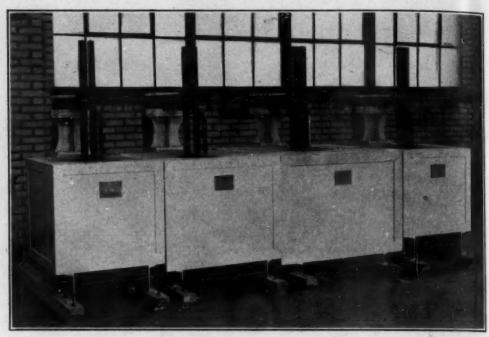


Fig. 14-Battery of Electrode Salt Baths for Heat Treating High Speed Steel.

pondingly larger, with accompanying heat losses, or the electrodes should be so disposed in the bath that the current has no occasion to pass through the main part of the bath containing the work to be heated. This means placing the electrodes at the side of the crucible in one or more pairs, the distance within each pair being small as compared with the distance from the electrodes to the work.⁵ Figs. 12 and 13 show two examples of this arrangement.

The arrangement described provides an additional advantage in that an electromagnetic stirring action may be produced in the salt as demonstrated in the Ajax-Hultgren furnace developed by the Ajax Electric Company for carburizing and other liquid heat treating operations at moderate temperatures. Thus, in spite of the heat being generated at one or more spots at the side of the crucible, a practically uniform temperature may be obtained throughout the bath. An installation of this type for hardening high speed steel comprising two preheat, one high heat and one quenching bath is shown in Fig. 14.

⁵U.S. patent number 1,811,754.

December

STUDY OF IRON OXIDE AND GAS CONTENT OF MOLTEN ALLOY STEEL

By Weston A. Hare and Gilbert Soler

Abstract

This paper presents the results secured by analysis of the gases evolved from molten alloy steels to determine the per cent FeO and hydrogen present at various stages of electric furnace and open-hearth heats. The gases found and their amounts, as well as the effect of various slags on these gases are discussed. The effect of moisture in the furnace atmosphere is also discussed, as is the effect of time on the pick-up of FeO under a nonreducing slag. Data are presented on the reduction of FeO and hydrogen effected by rabbling.

In order to meet these somewhat detailed specifications, it is necessary to have as accurate control as possible of the various factors involved in the melting and refining operations. One of the most important of these factors is a knowledge of the degree of oxidation (per cent FeO) of the steel bath during the working and devoxidation stages of a heat. On this factor depends the rate of loss of carbon and the loss of deoxidizing alloys such as manganese, silicon, and aluminum, so that some knowledge of the degree of oxidation of a bath of steel is useful even to meet the chemical specifications.

Much more intimately, however, the degree of oxidation of the steel bath is connected with a satisfactory control of grain size, hardenability, abnormality, etc. As is commonly recognized, and indicated by Herty¹ in his extensive work on deoxidation in the open-hearth process, the proper control of grain size requires a very careful regulation of the degree of oxidation in the final stages of the heat. It is also known that the inherent hardenability of

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¹C. H. Herty, "The Physical Chemistry of Steel Making," 1934.

Of the authors, Weston A. Hare is research engineer and Gilbert Soler is manager of research, Steel and Tube Division, The Timken Roller Bearing Co., Canton, Ohio. Manuscript received August 17, 1937.

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higher carbon steels may be changed considerably within the same analysis range by a variation in the deoxidation practice involving the variation of per cent FeO in the later stages of the heat.

The importance of control of oxidizing conditions throughout a heat of steel is readily confirmed by the present extensive use in steel melting of "Iron Oxide Slag Control." The control of the state of oxidation of a steel bath has generally been quite empirical, due to lack of a rapid and dependable means of measuring the percentage of iron oxide in the molten steel.

Of the several methods which have been available for obtaining per cent FeO in liquid steel, the Bureau of Mines method devised by Herty² and associates is most widely used. This has proven valuable, but it cannot be used as a control test due to the considerable time which must elapse before the chemical analyses are completed. It is also subject to certain errors, and for this reason requires special care in taking the test, drilling, and analysis. Moreover, tests taken after aluminum deoxidation may be in error due to residual Al₂O₃ inclusions.

Schenck³ has outlined an indirect method for determining per cent FeO by the rate of carbon drop. We have never been able to show this method to be applicable to the steels with which we are concerned, although there is certainly a rough relation between the per cent FeO and carbon at certain stages of a heat. Other factors, however, such as composition of slag, basicity and maturity of slag, variation of manganese and other elements in the bath, as well as the temperature, exert a large effect.

Much of the present control of the oxidation of a steel bath is obtained by a rapid analysis of the slag FeO on the assumption that the per cent FeO in the steel is roughly proportional to the FeO in the slag. This may be shown to be only approximately true, however, since many factors of slag composition, temperature, etc., affect the relation FeO steel/FeO slag, even when equilibrium relations between the two phases are attained.

Desiring to obtain a more exact knowledge of the per cent FeO in the steel bath, a method4 was developed at the Timken plant and recently reported which has allowed us a greater control of this phase of steel melting than has been previously possible.

²C. H. Herty et al, Bureau of Mines Report, 3166, 1932.

³Stahl und Eisen, Vol. 53, 1933, p. 1049. ⁴Hare, Peterson and Soler, "An Improved Method for Determining Gas Content of Molten Steel," Transactions, American Society for Metals, September 1937, p. 889.

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as Content of p. 889. This method consists of drawing a sample of molten steel from a sampling spoon into a specially designed evacuated tube and of analyzing the evolved gases volumetrically. This not only measures the gases as such evolved from molten steel, but has also been shown to measure with satisfactory accuracy the FeO present in the steel sample. The dissolved FeO reacts with dissolved carbon as follows:

 $Fe + C \rightarrow CO + Fe$

and inasmuch as the reaction has been shown to go very largely to completion under the conditions of the test, the weight percentage of carbon monoxide obtained may be calculated directly to per cent FeO. As discussed in the reference, the values of FeO obtained by this method have been found to check with fair accuracy values obtained by the Bureau of Mines Aluminum Method for FeO in steel. The speed with which per cent FeO in a steel bath may be obtained by the gas method would permit its use as a routine control test if desired.

We propose in the present paper to discuss more fully the usefulness of the method and illustrate the results obtained on various types of alloy steel.

The gases evolved from freezing steel have been subject to frequent study, but in the past, most methods of studying them have been subject to considerable error due to contamination with atmospheric gases or loss of a large portion of the gases. This difficulty has been largely due to a lack of a suitable sampling device. Those who have previously studied the subject report in general that carbon monoxide, hydrogen, nitrogen, and lesser amounts of carbon dioxide, methane, and oxygen are found in gases evolved from steel. Assuming that the gases are in thermal equilibrium, however, it is clear that some of these gases cannot be present to any large extent. Thus, only traces of oxygen could be present simultaneously with large amounts of either hydrogen or carbon monoxide and the ratio to be expected between CO₂ and CO is fixed by thermodynamic relations. Many of the results reported in the literature show relative amounts of these gases which are inconsistent with the equilibrium relations, probably due to contamination of the evolved gases with air.

The gases which we have obtained from molten steel consist almost entirely (over 95 per cent by volume) of carbon monoxide and hydrogen, the relative amounts of the two gases varying widely, depending on the degree of oxidation of the heat. In addition to

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these gases, a small amount of carbon dioxide is present, roughly the amount to be expected from equilibrium relations. A small amount of hydrocarbon gases, probably methane, is generally found. Oxygen is never found in appreciable amounts. Nitrogen, which might be expected as a gas evolved from freezing steel, frequently noted in prior work, has almost never been obtained in amounts exceeding 1 or 2 per cent. Most of our tests show either none or only small fractions of one per cent. Due to the long exposure of molten steel to the nitrogen of the atmosphere and the fact that it is generally considered as soluble in liquid steel, it is rather surprising that such small percentages are evolved. Evidently any nitrogen gas absorbed by the steel in open-hearth or electric furnace processes is largely held as a stable nitride.

RESULTS OF GAS ANALYSES OF MOLTEN STEEL

A set of typical results of analyses for various types of steel and various stages of the working of the heats is listed in Table I. These heats have been analyzed for CO and H₂ only. On special tests it is customary to report the other gases which are present in very small amounts. It may be noted that during the periods of working before any deoxidation, i.e., before reboil on the openhearth or before slag-off on the electric furnace, the CO and FeO values are relatively high and in general the hydrogen somewhat lower than after deoxidizing operations.

The value of FeO at reboil does not show in general any correlation with the carbon content as indicated in the case of the examples of S.A.E. 1020 and 1065 specification. We have found that although at earlier stages of the heat the per cent FeO in the steel is regulated by the per cent carbon present, this is not usually the case by the time the heat is ready for reboil. It appears to us that manganese or silicon generally control the per cent FeO by the time this stage is reached.

The values for per cent FeO in the steel at tap will be seen to be higher in the open-hearth heats than in the electric furnace as is to be expected in view of the slag conditions of the two furnaces. After ladle deoxidation, there is relatively little difference in the gas content of the steel from the two types of furnaces.

The diminishing of per cent FeO as the preliminary and final deoxidation proceeds will be seen in the open-hearth tests, particularly the S.A.E. 1065 heat. Having 0.13 per cent FeO at the time

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of reboil, this is gradually reduced by successive deoxidation to 0.062 per cent at tap, and by ladle deoxidation to 0.015 per cent at the time of teeming.

The same process occurs by a different method in the electric furnace heats. While before the first slag is removed, the FeO is similar to that found in the open-hearth, the addition of the reducing carbide slag cuts this per cent FeO to a fairly low value by tapping time, even in cases where no furnace deoxidation is used.

Table I
Typical Gas Analysis from Molten Steel

Furnace	S.A.E. Spec.	Time of Test	Per Cent H2	Per Cent CO	Calc. Per Cent FeO
Open-Hearth	1020	Before Reboil At Tap*	0.00070 0.00090	0.054 0.018	0.138 0.046
	1065	Before Reboil At Kill At Tap* Ladle Test	0.00067 0.00052 0.00068 0.00065	0.051 0.049 0.024 0.006	0.130 0.125 0.062 0.015
	4615	At Tap*	0.00090	0.016	0.041
Electric	4615	Bef. Slag-off At Tap Ladle Test	0.00030 0.00074 0.00040	0.038 0.014 0.007	0.098 0.036 0.018
	10105	Bef. Slag-off Prelim. Test† 45 min. after slag-off At Tap†	0.00110 0.00062 0.00165	0.054 0.009 0.020	0.138 0.023 0.051
	51100	Bef. Slag-off At Tap	0.00050 0.00100	0.037	0.095 0.025
	10150	Tap Ladle Test	0.00070 0.00037	0.005 0.003	0.013 0.007
	18-8 Stainless	Тар	0.00037	0.003	0.007

In the case of the S.A.E. 10105 heat it will be noted that the per cent FeO at "prelim" was considerably lower than at tapping time. This is due to the fact that in this particular heat the carbide slag was highly reducing and black in color at "prelim" time, but was allowed to "whiten" or lose its reducing qualities at tap. The difference between per cent FeO under a "black" carbide slag and a less reducing "white" carbide slag is thus indicated.

The hydrogen content of the steel in the samples of Table I are between 0.00030 and 0.00100 per cent. This is a usual range. However, higher values are frequently obtained. The amount of hydrogen seems to vary from time to time; whether with the amount of moisture in the atmosphere or otherwise we cannot as yet say. Thus we have averaged the per cent H₂ found in our tests of open-

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hearth steel at tap over a period of a year, and find a variation of per cent H₂ which seems to be seasonal in nature.

	Per Cent	Ha (Average)
December 1935, January, February 1936	24 tests	0.00066
March, April, May 1936	24 tests	0.00090
June, July, August 1936	31 tests	0.00093
September, October, November 1936	48 tests	0.00078

An example illustrating how hydrogen may be absorbed from a moist atmosphere is shown in the following set of tests. Two heats of electric furnace nickel-molybdenum steel were analyzed for gas content before slag-off and at tap. It was observed that a leak had developed in a cooler coil in the furnace, permitting water to enter the furnace throughout the entire heat. The results of gas analysis of these two heats compared with a heat of normal gas content at the time studied were as follows:

Heat	Time of Test	Per Cent Ha	Per Cent CO	Calc. Per Cent Fe0
27266	Before slag-off	0.00085	0.059	0.149
(Leak in cooler)	At Tap	0.00780	0.018	0.045
(Leak in cooler)	Before slag-off	0.00034	0.056	0.142
	At Tap	0.00650	0.017	0.043
37927	Before slag-off	0.00057	0.057	0.145
(Normal heat)	At Tap	0.00060	0.007	0.018

The effect of the leaking water was not noticed at slag-off since the oxidized condition of the bath or the effect of the "boil" probably precluded the presence of much hydrogen gas. Under the reducing second slag, however, the hydrogen content rose to a value nearly ten times the usual value. It would be reasonable to suppose that this excess hydrogen should affect the soundness of the steel or its physical properties. No such effect was observed in either the transverse etch or other properties studied, so that the hydrogen presumably escaped during tapping and pouring.

PICK-UP OF FEO WITH TIME UNDER NONREDUCING SLAG

An interesting study was made on high carbon-chromium (S.A.E. 52100) electric furnace steel in a series of experimental heats employing a second slag without coke addition. The heats after removal of the first slag were finished under a slag of lime and sand of less than 2:1 CaO-SiO₂ ratio. Tests were taken for FeO by the gas method before deoxidation additions near tap. The variation of per cent FeO obtained was correlated with the time under the second

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slag, as illustrated in Fig. 1. It will be noted that there is a regular increase in the per cent FeO with time under these conditions, averaging about 0.0025 per cent FeO per minute. Thus heats tested

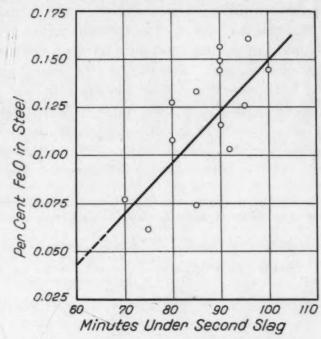


Fig. 1-Per Cent FeO by Gas Analysis Versus Time Held Under Nonreducing Second Slag.

after one hour and ten minutes averaged around 0.070 per cent, while those held as long as one hour and forty minutes would contain approximately twice this amount of dissolved iron oxide. It must be noted, of course, that the slag in these heats was not made reducing with coke as in the usual electric furnace practice. Had this been done, no such increase of per cent FeO with time would be observed.

It is often the practice in electric steel manufacture to rabble the heat while under the carbide slag. This is done by a heavy iron bar which is used to stir the steel, producing a boil on the bar and effecting thorough mixing of the bath. Several sets of tests on S.A.E. 52100 steel were taken to note the effect of this "rabbling" on the per cent FeO and gas content of the bath.

			Gas Con	ntent-	
Heat	Time of Rabbling	Per Cent H ₂	Per Cent FeO from CO	After R	Per Cent Fed from CO
1	At Prelim.	0.00090	0.046	0.00060	0.035
1	At Tap	0.00050	0.038	0.00064	0.031
2	At Tap	0.00035	0.076	0.00065	0.033
3	At Tap	0.00062	0.054	0.00043	0.028
4	At Tap	0.00110	0.066	0.00105	0.064

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It will be seen that in general the per cent FeO in this steel is dropped by the use of the rabble and that the per cent hydrogen is somewhat irregular. It is probable that the effect of rabbling is to eliminate any supersaturation of FeO which might be expected to occur under a carbide slag due to the lack of action in the bath. The practice of thus eliminating supersaturation of FeO is certainly desirable for best quality steel. The use of any neutral gas bubbled through the steel bath causes a similar reduction of per cent FeO. Along this line some experiments were made in which carbon monoxide, nitrogen, etc., were bubbled through the bath by means of a steel pipe immersed in the molten metal. Compressed gases from tanks were used. The results obtained are shown below:

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			-Gas Conte	nt of Steel-	E INTE
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		1	Per Cent Fel) P	er Cent FeO
Gas Used	Test No.	Per Cent H ₂	from CO	Per Cent H ₂	from CO
0 1 36 11		0.00125	0.000	0.00080	0.000

			er Cent FeC		er Cent FeO
Gas Used	Test No.	Per Cent Ha	from CO	Per Cent H2	from CO
Carbon Monoxide	1	0.00135	0.077	0.00070	0.038
Carbon Monoxide	2	0.00058	0.069	0.00053	0.062
Carbon Monoxide	3	0.00068	0.082	0.00060	0.074
Carbon Monoxide	- 4	0.00065	0.073	0.00057	0.065
Ammonia	5	0.00102	0.072	0.00085	0.059
Ammonia	6	0.00090	0.044	0.00090	0.044

In Test No. 6, 0.007 per cent nitrogen was found, but it is uncertain whether from a slight leak or from actual evolution from the steel after absorption from the NH_a.

In general, is it clear that both FeO and hydrogen were somewhat reduced by the gas treatment as by rabbling.

Conclusion

This work has demonstrated the value of gas analysis primarily as a research tool in setting up standards of furnace practice. We have found it particularly valuable in determining the effect of variations in melting practice on inclusion content, hardenability and other physical properties of alloy steels. When a suitable furnace practice has been developed, gas analysis need not be continued as a routine, but may be used as an occasional check. However, in the preparation of special heats or new analyses, the analysis of gas taken from the molten steel offers a rapid and effective method of quality control.

THE USE OF CHROMIC ACID IN ELECTROLYTIC ETCHING OF IRON AND STEEL

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By M. BAEYERTZ

Abstract

An electrolytic etching method is suggested in which the sample is made the anode and a 10 per cent aqueous solution of chromic acid is used as the electrolyte. This method of etching appears to have a fairly wide field of application both in ordinary and alloy steels and in the stainless iron-chromium and iron-nickel-chromium alloys. The etching procedure is described and illustrations of several applications of this method of etching are given.

THE use of chromic acid in the electrolytic etching of steel and iron has a rather wide field of application in revealing various microstructures of these materials. The suggested etching procedure is an electrolytic method in which the sample to be etched is made the anode and an aqueous solution of chromic acid is used as the electrolyte. Under these conditions cementite and also the chromium carbides of the stainless steels are attacked rapidly, austenite is attacked more slowly, and ferrite still more slowly if at all.

The details of the suggested etching procedure follow. The electrolyte is prepared by dissolving 10 grams of CrO₃ in 100 milliliters of distilled water. The current is supplied by four dry cells in series or equivalent six volt source. The sample to be etched is made the anode and a flat piece of platinum the cathode. The distance between the surface to be etched and the cathode is kept between 3/4 and 1 inch. The length of time used for the etching depends on the structure to be developed. All specimens shown in the photomicrographs in this paper were etched by the above procedure and the etching time used is noted with each photomicrograph.

The rapid attack on cementite is illustrated by the piece of grey cast iron shown in Fig. 1. While cementite is rapidly attacked, ferrite and iron phosphide are evidently not attacked. By following the chromic acid etch with a proper etch in nital it is possible to differentiate iron phosphide, cementite and ferrite. The cementite is

The author, M. Baeyertz, is assistant research engineer, Carnegie-Illinois Steel Corp., South Works, Chicago. Manuscript received September 16, 1936.

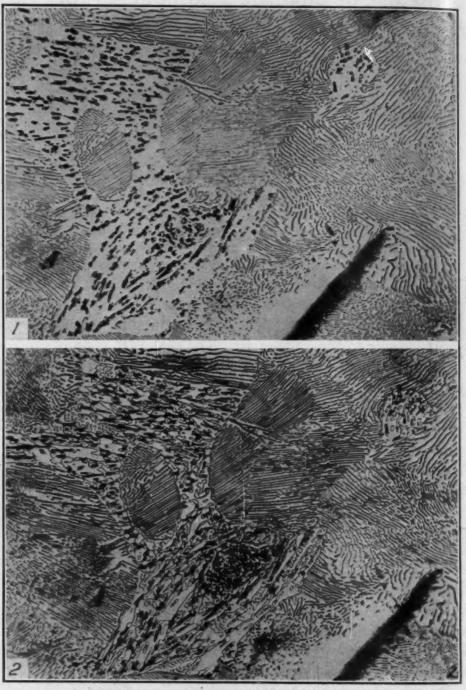


Fig. 1—Grey Cast Iron, Electrolytic Chromic Acid Etch, 60 Seconds. × 500. Fig. 2—Same Area as Fig. 1, Electrolytic Chromic Acid Etch Followed by 30 Seconds Etch in 2 Per Cent Nital. × 500.

first deeply etched by the electrolytic chromic acid method leaving the phosphide and ferrite unattacked (see Fig. 1, etched for 60 seconds). This etch was followed by etching in 2 per cent nital for 30 1937

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seconds (see Fig. 2). By this latter etch the ferrite is attacked, but not as deeply as the cementite had been by the electrolytic etch. The phosphide, which is not attacked by either the electrolytic chromic acid etch or by nital, stands out in relief above the ferrite.

The attack of the electrolytic chromic acid etch on cementite in a plain carbon steel is illustrated by Fig. 3. This steel was carburized at 1700 degrees Fahr. and slowly cooled after carburizing. The

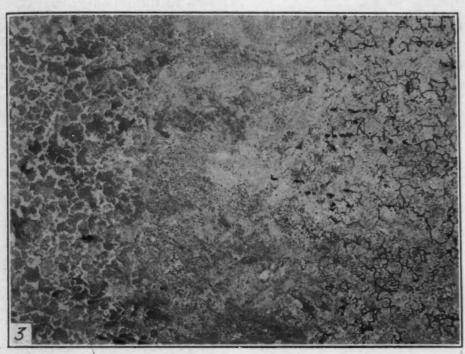


Fig. 3—Carburized Test on Plain Carbon Steel: Hypoeutectoid Zone (Left), Eutectoid Zone (Genter), and Hypereutectoid Zone (Right). Etched for 90 Seconds.

hypereutectoid zone is shown at the right, the eutectoid zone in the center, and the hypoeutectoid zone at the left. The cementite in the grain boundaries in the hypereutectoid case and the cementite lamellae of the pearlite have been attacked and hence appear dark in the low magnification photomicrograph. It is evident from Fig. 3 that the austenite grain size of both hypereutectoid zone and hypoeutectoid zone or core can be easily determined after etching by this method. This method of etching is also satisfactory for determining the extent of decarburization in pearlitic structures, since the lack of attack on ferrite leaves the ferrite as white areas unbroken by any etching of the grains or grain boundaries.

The electrolytic chromic acid etch is also adapted to the etching

of spheroidized cementite for examination at higher magnifications. After a light etch the carbide particles appear slightly depressed below the surface of the ferrite. The attack by this etching procedure is exactly opposite, in the case of ferrite and cementite, to the usual nital or picral etch in which the ferrite is attacked leaving the cementite particles standing in relief above the level of the ferrite. However, because of the different rate of attack, depending on the orientation of the ferrite grains exposed to the etching reagent, the relief of the cementite particles above the ferrite differs in differently oriented grains of ferrite when such structures are etched with nital. With the electrolytic chromic acid etch, since cementite but not ferrite is attacked, only those particles in the plane exposed to etching are revealed and the appearance of the cementite is not affected by differences in the orientation of the ferrite grains.

Since the electrolytic chromic acid method primarily attacks cementite, the etching of quenched and tempered martensites was observed. A series of S.A.E. 1045 steel test pieces, as quenched in water and tempered at increasing temperatures, are shown in Figs. 4 to 7, inclusive. The first tempering of martensite is brought out very plainly by the electrolytic chromic acid etch. The first tempering of martensite as indicated by the electrolytic chromic acid etch coincides with the first drop in Rockwell "C" hardness. The hardness values have been recorded in the caption for each tempering temperature.

The tempering of martensite in a hypereutectoid steel is shown in Fig. 8 which illustrates the hypereutectoid case of carburized S.A.E. 4615. This steel was carburized at 1700 degrees Fahr., quenched in oil from the carburizing box and then tempered at 450 degrees Fahr. The photomicrograph shows the plates of martensite which have decomposed at 450 degrees Fahr. to form a fine dispersion of cementite in ferrite. The remaining regions (white in the photomicrograph) are either undecomposed austenite or martensite in which the cementite particles are too small to give the surface a dark appearance under the microscope after attack by the electrolytic chromic acid etch.

Another example of satisfactory delineation of structure by the electrolytic chromic acid etch is illustrated by Fig. 9. This figure shows lighter etching and darker etching decomposition products of austenite developed on air cooling a ½ x ¼ x 4-inch piece of S.A.E. 5150 steel from 1830 degrees Fahr.

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S.A.E. 1045 Steel Quenched in Water and Tempered for 1 Hour, All Etched for 90 Seconds. × 1000.

Fig. 4—Tempered at 210 Degrees Fahr., Rockwell C = 59 (As Quenched = 59).

Fig. 5—Tempered at 300 Degrees Fahr., Rockwell C = 58.

Fig. 6—Tempered at 400 Degrees Fahr., Rockwell C = 56.

Fig. 7—Tempered at 650 Degrees Fahr., Rockwell C = 50.

This etch also reveals undiffused concentration and rarefaction of carbon in austenite (martensite after quenching) which correspond to the lamellae of the pearlite from which the austenite was

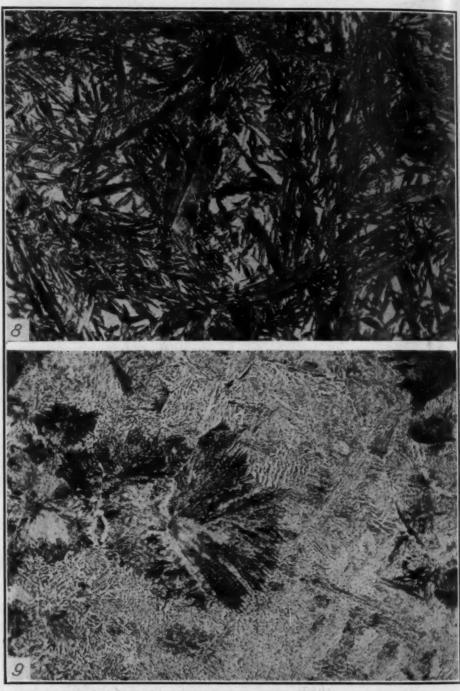


Fig. 8—Hypereutectoid Case—S.A.E. 4615 Steel Quenched in Oil and Tempered 1 Hour at 450 Degrees Fahr., Etched for 90 Seconds. × 500.

Fig. 9—S.A.E. 5150 Steel Air-cooled from 1830 Degrees Fahr., Etched for 2 Minutes. × 500.

derived. This is illustrated by Fig. 10 which shows the structure of a 1.10 per cent plain carbon steel after quenching from just above the Ac₁. In Fig. 10 the grain boundaries are in general outlined with

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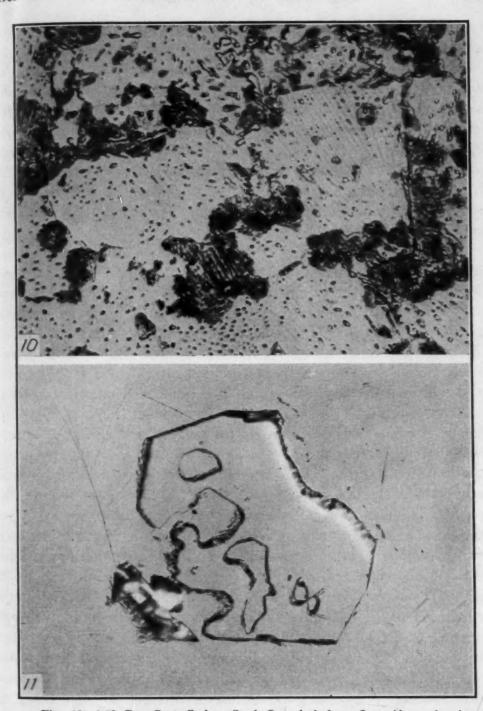


Fig. 10—1.10 Per Cent Carbon Steel Quenched from Just Above the Ac₁, Etched for 90 Seconds. × 1000. Fig. 11—27 Per Cent Chromium Steel, Etched for 10 Seconds. × 500.

cementite particles; considerable nodular pearlite is present near the grain boundaries and some within the grains. The rest of the structure consists of martensite with small etched-out globules or rods

which indicate the undissolved cementite remnants of the cementite lamellae of the original pearlite. Corresponding to the positions of these small particles of undissolved cementite, there is also a lamellar structure in the martensite and nodular pearlite which represents carbon concentration and rarefaction inherited from the pearlite before its transformation to austenite and again inherited by the martensite and nodular pearlite which were formed when the sample was quenched. When etched with nital these martensitic areas show the Widmannstätten pattern characteristics of martensite with no indication of the "cementite ghosts" which are brought out by the electrolytic chromic acid etch.

The chromium carbides in the iron-nickel-chromium and ironchromium stainless alloys such as 18 per cent chromium-10 per cent nickel, 25 per cent chromium-12 per cent nickel, 17 per cent chromium and 27 per cent chromium are rapidly attacked by the electrolytic chromic acid etch. The attack on the carbide in a large bloom of 27 per cent chromium steel is illustrated by Fig. 11. This photomicrograph shows the sharp preferential attack on a large carbide crystal leaving the ferrite in which it is embedded untouched. It is evident from Fig. 11 that the carbide crystal is below the level of the surrounding ferrite whereas in the polished and unetched sample these carbide crystals are always either level with or slightly above the level of the ferrite. By continuing the etching for a longer time such carbide crystals are entirely removed, leaving a hole in the ferrite. The attack of carbide in the grain boundaries of an 18 per cent chromium-10 per cent nickel alloy is shown in Fig. 12. Etching for 10 seconds is sufficient to outline the carbide-containing portions of the grain boundaries, while a somewhat longer time (up to 90 seconds) was required to delineate those portions of the grain boundaries which contained no carbide and to bring out the twin structure of the austenite. The attack on the austenite is less rapid than the attack on the carbide and its rate also depends on the orientation of the austenite grain or twin with respect to the surface exposed to the etching. Thus the grain and twin structure, in the absence of carbide, is revealed by differences in level of each grain or twin with respect to adjacent grains or twins. As shown by Figs. 13 and 14, this method of etching not only reveals the grain and twin structure of the austenite but also shows the presence of ferrite (stringers in Figs. 13 and 14) without overetching the ferrite. This is due to the fact that the electrolytic chromic acid etch attacks the ferrite much

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Fig. 12—18 Per Cent Chromium-10 Per Cent Nickel Steel with Carbide in Grain Boundaries, Etched 60 Seconds. × 500.

Fig. 13—19.66 Per Cent Chromium-11.35 Per Cent Nickel-0.02 Per Cent Carbon Steel Showing Both Ferrite and Inclusions, Etched for 90 Seconds. × 100.

more slowly than it attacks the austenite. Hence the grain and twin structure of the austenite is developed by attack, leaving the ferrite standing in relief above the austenite (see Fig. 14). This is the oppo-

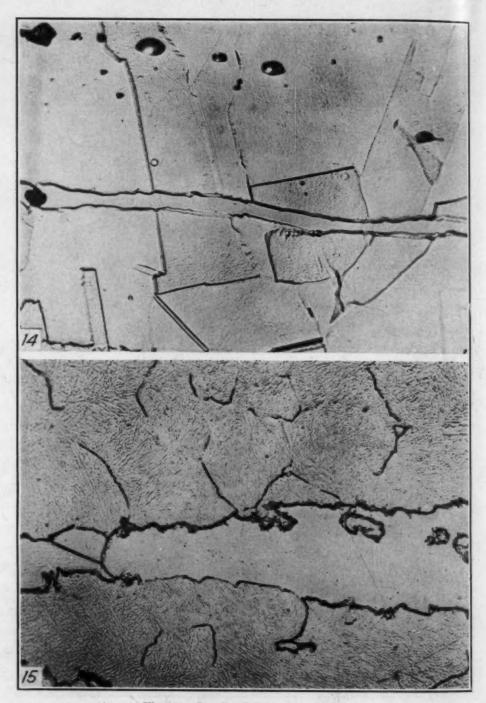


Fig. 15—17 Per Cent Chromium Steel, As-Rolled, Etched for 30 Seconds. × 500.

site effect to that obtained by some of the usual methods of etching these alloys (aqua regia both alone and modified by the addition of acetic acid and glycerine, and the electrolytic oxalic acid etch¹) by 1937

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¹G. A. Ellinger, Transactions, American Society for Metals, Vol. 24, March 1936, p. 26-35.

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which the ferrite is attacked more rapidly than the austenite. This characteristic of the electrolytic chromic acid etch permits small stringers of ferrite to be readily distinguished from inclusions even after the sample has been etched deeply enough to reveal the grain and twin structure of the austenite (see Fig. 13). The alloy in Figs. 13 and 14 contained 19.66 per cent chromium, 11.35 per cent nickel and 0.02 per cent carbon.

The martensite and ferrite structure in 17 per cent chromium steel as etched by the electrolytic chromic acid etch is shown in Fig. 15. The etching of this structure is apparently due to attack on precipitated carbides in the martensite and along grain or phase boundaries.

Besides attacking cementite and chromium carbides very rapidly, the electrolytic chromic acid etch also rapidly attacks the manganous sulphide-ferrous sulphide solid solution customarily observed in ordinary steels. It also attacks the sulphides in the iron-nickelchromium and iron-chromium stainless alloys. This latter is not the case, at least in our experience, when the sulphides in these stainless alloys are subjected to 10 per cent aqueous chromic acid solution (Campbell-Comstock scheme for identification of inclusions²) without using this reagent electrolytically. The failure to obtain attack on the sulphides in these stainless alloys on simple immersion in chromic acid solution may be due to failure of the etchant to wet the polished surface of the sample or to variation in the composition of these sulphides which makes them less susceptible to attack by this etchant. However, by using a 10 per cent aqueous solution of chromic acid as an electrolytic etchant and making the sample the anode, rapid attack of the sulphides in these alloys is obtained. This is illustrated by Figs. 16 and 17, before and after etching respectively. In Fig. 17 the light grey sulphides, present in Fig. 16, have been completely removed by 10 seconds etching. The inclusion at the top of Fig. 16 contains three mineral constituents: sulphide, light grey; chromite, medium grey; and silicate, very dark grey. It is evident from Fig. 17 that of these only the sulphide is attacked. It may also be noted in passing, since titanium is sometimes added to these alloys, that titanium nitride inclusions are not attacked by the electrolytic chromic acid etch. An interesting effect was observed on an 18 per cent chromium-10 per cent nickel-0.26 per cent selenium stainless steel. This steel contains numerous inclusions which in the unetched

²A.S.M. Metals Handbook, 1936 edition, p. 561.

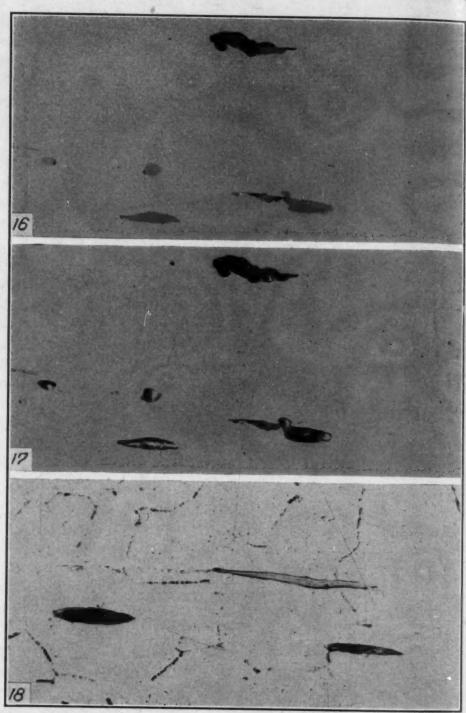


Fig. 16—Sulphide, Chromite and Silicate in 17 Per Cent Chromium-10 Per Cent Nickel Steel, Unetched. × 500.

Fig. 17—Same Area as Fig. 16, Etched for 10 Seconds. × 500.

Fig. 18—18 Per Cent Chromium-10 Per Cent Nickel-0.26 Per Cent Selenium Steel, Etched for 60 Seconds. × 500.

state correspond in color and appearance to the sulphides observed in regular 18 per cent chromium-10 per cent nickel alloy (see Fig. 16).

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However, on etching by the electrolytic chromic acid method, some of these inclusions are attacked much more rapidly than others (see Fig. 18). Since the sulphides in regular 18 per cent chromium-10 per cent nickel are rapidly attacked, it seems reasonable to assume that, in selenium-bearing 18-10, the inclusions rich in selenium are attacked more slowly while those rich in sulphur are attacked more rapidly.

The suggested electrolytic chromic acid etch attacks cementite and chromium carbides of stainless steels very rapidly, austenite less rapidly and ferrite very slowly if at all. Iron phosphide is likewise not attacked. The customary manganous sulphide-ferrous sulphide solid solution in ordinary steel and the sulphides in the iron-chromium and iron-nickel-chromium stainless alloys are rapidly attacked. The selenides in selenium-bearing 18 per cent chromium-10 per cent nickel are much less rapidly attacked than the sulphides. Titanium nitride, chromite and a silicate (possibly not all silicates) are not attacked.

THE EFFECTS OF VARIATION IN DIAMOND INDENTERS USED IN THE VICKERS HARDNESS TESTING MACHINE

By F. B. FULLER

Abstract

The Vickers hardness testing machine, developed a few years ago in England for the testing of thin sheet, has been receiving more and more consideration in this country in the study of surface-hardened materials, of welded joints and of thin sheet material. The comparison of hardness data from laboratories may be seriously affected by the diamond indenter procured from different sources and this paper is presented for the purpose of calling attention to the variations that may be found in diamond indenters obtained from different sources rather than for the purpose of discussing testing procedure.

This paper describes the hardness data obtained with a Vickers hardness machine using three individual "standard" diamond pyramid indenters under comparable testing conditions and on uniformly hardened alloy steel blocks over a wide range of hardness. The results indicate that discrepancies in hardness data and nongeometrically shaped impressions may be expected for properly calibrated and operated Vickers hardness machines unless the accuracy and workmanship of the diamond pyramid indenters are satisfactorily controlled.

THE Vickers hardness testing machine was developed in England and has been standardized there. It is based upon the principle of reading the two diagonals of a square impression produced by a sharp pointed pyramid diamond indenter, having an included angle of 136 degrees between opposite faces. The indenting load, ranging from 5 to 100 kilograms as desired, is applied at an even rate through a system of levers and dead weights. The diagonals of the resulting square impression are measured by means of a micrometer ocular microscope, capable of being rotated 90 degrees, and containing a

¹Hugh O'Neil, "The Hardness of Metals and Its Measurement," p. 88, The Sherwood Press, 1934.

Note—Within the past year a diamond indenter, imported from England, has been purchased from a domestic source and was found to be in good agreement with the original one obtained with the machine.

The author, F. B. Fuller, is Materials Testing Engineer, Materials Branch. U. S. Army Air Corps, Wright Field, Dayton, Ohio. Manuscript received February 3, 1937.

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Table I Hardness Values (Vickers Pyramid Numerals) Obtained with Pyramid Diamond Indenters

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Hardness data on a nitrided specimen with diamonds No. 1 and 2 under a 10-kilogram load made previously to this investigation were 1048 974 respectively. NOTE: Steel No. 1 and 5-Cyclops. Steel No. 2 and 4-S.A.E. 6140. Steel No. 3-S.A.E. 3130. Steel No. 6-S.A.E. 1045. and

*Two hardness numbers per single spot, taken at 90 degrees to each other, for all the values in columns headed "single spots."

throughout this work.

fixed and a movable knife edge. The ocular readings taken are converted into Vickers Pyramid Hardness Numerals obtained from charts received with the machine. One digit change in the ocular number as read with the ½3 objective corresponds to a change in lineal dimension of 0.002 millimeter. The ½3 objective was used

The machine at Wright Field was equipped with a diamond pyramid indenter when received from England approximately four years ago. This indenter is No. 1 in Table II.

Table II

Angular Measurements of Pyramid Diamond Indenters

Diamond No.	-Included	l Angles*	Average for Included		ntary Angl	es of Includ	ed Angles
1 2	136° 40′ 145° 6′	137° 16′ 144° 30′	Angles 136° 58' 144° 48'	68° 36′ 72° 4′	68° 4′ 73° 2′	68° 16′ 71° 36′	69° 0′
3	142° 0′	142° 0'	142° 0′		13-2	71-36	72° 54′

*The specified included angle for a Vickers Pyramid Indenter is 136°.

An indenter obtained recently from a domestic firm was returned due to the extremely "lopsided" impression produced. A second indenter received from the same source, and a third one furnished by another domestic manufacturer, have also proven unsatisfactory. It is understood that other laboratories are having the same difficulties and the results obtained on different Vickers hardness machines do not check. This information and the preliminary data from the three indenters led to a more detailed study.

Materials. Hardness tests were made on hardened alloy steel blocks, $1 \times 1 \times 3$ inches, Table I. A pair of opposite faces were then ground parallel with a 38 alundum grinding wheel. Approximately $\frac{1}{16}$ inch was removed from the test face, which was then finished on a belt sander using No. 100 grit.

The designations used throughout this paper and the history of the diamond indenters are as follows:

Diamond No. 1. Original as received from England with the Vickers machine.

Diamond No. 2. Submitted by a domestic firm about November 1934, in lieu of one which had been returned to them as unsatisfactory, the impression being "lopsided."

Diamond No. 3. Submitted about January 1935, by another domestic firm, reputed to be high-grade diamond grinders.

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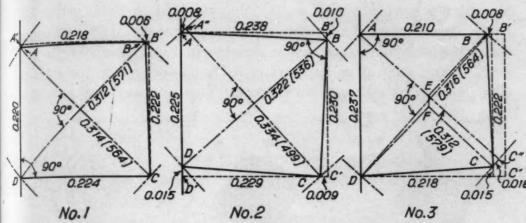


Fig. 1—Measurements on Impression Made with Pyramid Diamond Indenters in Vickers Hardness Testing Machine with 30-Kilogram Load on Rockwell C-53 1045 Steel Block.

TESTING PROCEDURE

These data for comparative purposes were Hardness Results. obtained from two impressions made with each indenter in the center of each test block, grouped closely together but with sufficient distance to eliminate interference. Loads of 10 and 30 kilograms were used. Two hardness readings were taken on each impression, the first between one pair of opposite corners and the second between the other pair of opposite corners after the micrometer ocular head had been rotated 90 degrees. Each diamond indenter was so oriented in the indenting plunger that lines through the two opposite corners to be measured were parallel to the fixed and movable knife edges. This was readily accomplished with Diamond No. 1 which had an index line inscribed on the piece in which it was mounted that was brought into coincidence with a line on the indenting plunger. It was necessary to determine this position for Diamond No. 2 and 3 by trial. The impression should be properly positioned with reference to the measuring head of the microscope when the indenter is properly set in the indenting plunger.

Loads of 10 and 30 kilograms have been used for practically all test work in this laboratory, the 10-kilogram load for nitrided and other surface hardened steels and on thin gage and soft material, and the 30-kilogram load for homogeneous materials.

Impression Measurements. The measurements for squareness (Fig. 1) were obtained on impressions made with a 30-kilogram load on an alloy steel block of Rockwell C-53. The photographs for surface outline (Fig. 2) and depth study (Fig. 3) were obtained

on impressions made with a 100-kilogram load in order to obtain better definition at the bottom of the impression. The procedure for developing the diagrammatic sketches in Fig 1 consisted of orientating one side of the impression, A.D., parallel to the fixed knife edge of the Vickers ocular microscope. This side was used as a ref-

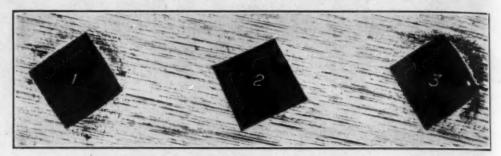


Fig. 2—Photograph of Surface Outline of Indentation Made with Pyramid Diamond Indenters in Vickers Hardness Testing Machine with 100-Kilogram Load on Rockwell C-53 S.A.E. 1045 Steel Block.

erence line and the microscope or the specimen rotated at 90-degree turns and ocular readings noted. These were then converted into millimeters from a calibration chart. The impressions were also examined at approximately 80 diameters with binoculars. The sur-



Fig. 3—Photograph of Depth Impression of Indentation Made with Pyramid Diamond Indenters in Vickers Hardness Testing Machine with 100-Kilogram Load on Rockwell C-53 S.A.E. 1045 Steel Block.

face of the block used in this part of the investigation was given a better finish to obtain a very definite outline.

Diamond Measurements. The included angles were measured on a Zeiss machine. Equipment was not available for checking the angle between each face and a plane passing vertically through the center of the pyramid.

Calibration of the Vickers Hardness Machine. The ocular measurements taken with the Vickers micrometer microscope showed

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good agreement when checked against a special micrometer measuring microscope reading direct to 0.01 millimeter. The loads were not calibrated directly, but the data obtained with the original diamond were found to be in accordance with previous data for the same loads. These two factors were not essential to this discussion but are presented to establish the accuracy of the machine.

The original diamond, Diamond No. 1, is used as the basis of comparison in all the work presented in this paper since the impressions are practically square and consequently the diagonals are approximately equal. The data in the included angle are in close agreement with that specified for a standard pyramid indenter, namely, 136 degrees. The fixed rotation of the ocular micrometer measuring head of the microscope was checked practically at 90 degrees.

RESULTS AND DISCUSSION

The hardness data in Table I are quite comparable throughout the range for Diamond Nos. 1 and 3 under the 30-kilogram load whereas for Diamond No. 2 they are considerably lower in the higher hardness range. The results with the 10-kilogram load for Diamonds No. 2 and 3 were obtained for the purpose of comparing with these for the 30-kilogram load. Check tests with Diamond No. 1 were not made since previous data with a 10-kilogram load had given results comparable to those for the 30-kilogram load.

There is also considerable more spread between the two hardness determinations for each impression in the higher hardness ranges for Diamond No. 2. One digit variation in ocular measurement in the higher ranges where the impressions are small results in a greater per cent variation in hardness units than in the lower hardness ranges where the impressions are relatively large. Therefore, any irregularities in the impressions produce greater differences in the upper range under the lighter loads where the Vickers machine should show the greatest advantage.

The factors of friction between the diamond faces and the metals and of "piling up" or "sinking in" of the metal around the impression are considered negligible in this paper in the study of the variation in the hardness values. The faces of the indenter were polished satisfactorily and there was only a very slight but comparable piling up around each impression on the steels tested. It must be recognized that determinations of hardness on the basis of surface measure-

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The included angles for Diamond No. 1, noted in Table II, measured very closely to that of 136 degrees specified for these indenters. Those for Diamond No. 2 and No. 3 were considerably greater. A comparison of the shape of the outlines as viewed on the surface is shown in Fig. 2 at approximately 50 diameters for the three impressions made with a 100-kilogram load. A diagrammatic picture of the same impressions with measurements is given in Fig. 1. The dark area noticeable around impression No. 3 (Diamond No. 3) in Fig. 2 is due to the lighting effect since examinations with the Vickers microscope and with binoculars indicate practically no irregularity in bulging or "piling up" around the impressions.

The impression on the surface made with Diamond No. 1 was practically square with equal measurements between opposite corners: that for No. 2 was "lopsided" with decidedly unequal measurements, and that for Diamond No. 3 was "lopsided" with practically equal measurements. The measurements for hardness are not true diagonals of a square except in the case of Diamond No. 1, but rather are measurements taken at right angles to each other when the specimen is orientated so that lines through two opposite corners are parallel to the measuring knife edges. The line A"C for Diamond No. 2 and AC" for Diamond No. 3 in Fig. 1 represents one line for a hardness determination. The broken outline A B' C' D represents the outlines for a square upon the basis of the largest measurement for that impression and the solid line A B C D represents the actual outline. In the case of a true square the hardness measurements at 90 degrees to each other, when the impression is properly positioned in the microscope, would represent true and consequently equal diagonals. The lines AE, BE, EF, DF and CF for Diamond No. 3 in Fig. 1 represent the appearance of the impression in depth.

The "lopsidedness" of the impressions of Diamond No. 2 and 3 indicate that either the faces are not ground so that they are properly aligned with each other or that the axis of the pyramid does not coincide with the axis of the indenter shaft.

A study of the characteristics at the bottom of the impressions in Fig. 3 show that the corner lines meet at a point and were coincident from two opposite corners for the impression made by Diamond No. 1. For Diamond No. 2 there is a slight offset of the corner lines indicating that the apex of the diamond was not ground to a sharp

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point. The bottom of the impression for Diamond No. 3 shows a marked offset. This offset or blunt chisel condition is noted in Fig. 1 by the lines AE, BE, EF, DF and CF, and it is evident from a study of the shape that the hardness measurements across corners might be practically equal irrespective of the "lopsided" outline. An examination of the apex of the diamonds with binoculars verified the presence of the "offsets."

The lower hardness values for Diamond No. 2 may be partially accounted for by the greater included angle, being approximately 9 degrees more than the specified angle of 136 degrees. Data by Smith and Sandland² indicate a decrease in hardness for a steel block of from 895 with a 136-degree pyramid diamond indenter to 856 for a 140-degree angle. The hardness data obtained with Diamond No. 3 which has an included angle greater than 136 degrees was not in agreement with these findings. The results were comparable to those for the original diamond probably due to a compensating effect of the blunt point which would develop greater resistance to penetration under the same load and indicate a higher hardness.

The tolerances permissible in the characteristics of the diamond indenter require more study. The measurements and the outline of the impression made with the original Vickers diamond indicate that careful workmanship will result in a diamond pyramid indenter that will produce practically a square impression. The measurements of the total included angle and of the two supplements of the included angle show close agreement with the specified 136-degree angle.

Conclusions

The British Standards Institution give the following qualifications for the Vickers diamond pyramid taken from B.S. Specification No. 427-1931.³

1(a). The diamond indenter shall be in the form of a square pyramid and have an angle between opposite faces of 136 degrees. The pyramidal faces shall have a good polish and be free from "pits" or other surface blemishes. The point of the diamond shall be sharp.

The specifications should be more specific and the following recommendations are made:

1. A tolerance should be stated on the included angle that is

²Loc. Cit., p. 41.

³Loc. Cit., p. 88.

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within the limits of careful and fine workmanship in diamond grinding.

- 2. The faces should be perpendicular to two 90-degree vertical planes passing through the axis of the pyramid which shall coincide with the axis of the indenter assembly.
- 3. An index line, similar to that on the original Vickers indenter, shall be inscribed on the mounting in which the diamond is set so that the impression is properly positioned with reference to the micrometer ocular microscope.
- 4. The diamond shall be mounted in a position in which a line from the apex extending through the middle of the pyramid base is parallel to the direction of the indenting load.
- 5. The impressions made with 10 and 30-kilogram loads on a hardness block of approximately 600 Brinell shall be square.

ACKNOWLEDGMENT

The author wishes to express his appreciation for the interest of Mr. J. B. Johnson, Chief, Material Branch, Wright Field, Dayton, Ohio.

X-RAY DIFFRACTION STUDIES OF DISTORTION IN METALS

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By George L. Clark and Merton M. Beckwith

Abstract

Specimens consisting of both single crystals and polycrystalline aggregates of aluminum were subjected to deformation by measured tension in successive steps and X-ray diffraction patterns made for each state of the specimen. A special specimen holder with which a measured tension could be communicated to the specimen through calibrated springs was designed so that it could be placed directly on the X-ray tube assembly. Throughout the whole range of tensile stresses from zero to fracture the X-ray beam passed through the same area in the specimen. In all cases the orientation of the crystals was determined for the original state. Several series of diffraction patterns are represented in the illustrations.

For a single crystal specimen the first change in Laue spots is a shadow appearing below the interference from the (111) planes. The successive changes in the diffraction interferences support the theory that the distortion in a single crystal is bending, followed by fragmentation, with subsequent preferred orientation of the crystal fragments. The series of patterns for polycrystalline aluminum show that the mechanism of distortion is the same for an aggregate of small crystals as for a single crystal. The change in each small spot is the same as the change for the identical spot from a single crystal. The final stage in deformation is fragmentation into a very large number of small crystals as shown by the production of smooth Debye-Scherrer rings. The rotation of the fragmented crystals is demonstrated by the formation of intensity maxima on the rings, indicative of preferred orientation.

THE distortion of metallic specimens has been of interest for a great many years. Hooke's law, that stress is proportional to strain, within the elastic limit, was enunciated in the seventeenth cen-

A paper presented before the Eighteenth Annual Convention of the Society held in Cleveland, October 19 to 23, 1936. Of the authors, G. L. Clark is professor of chemistry at the University of Illinois, Urbana, Ill., and M. M. Beckwith is associated with the Research Laboratory, Guide Lamp Company, Anderson, Ind. Manuscript received July 25, 1936.

tury. From that time on down to more recent years, there has been much speculation concerning the elasticity and the mechanism of failure of a metal. During most of this time only scattering data were collected and poorly grounded hypotheses presented.

After the microscope was first applied to metallurgical problems, data and information became available which shed some light on many of the phenomena known at that time. Using the microscope, Ewing and Rosenhain (1)¹ were the first to observe the appearance of a series of lines on the exposed surface of a metal crystal, when the aggregate mass, of which it was a member, had been deformed. They gave these lines the name of "slip lines." Using the microscope other investigators confirmed these findings.

The next great scientific advance which affected the study of distortion was the discovery, in 1912, by von Laue, that X-rays were diffracted by crystals.

There are two types of patterns produced by the diffraction of X-rays by a crystal grating. If a single crystal is used with a polychromatic beam, the resultant pattern is made up of a series of diffraction interferences in the form of spots. If the crystal is arranged with a crystallographic axis parallel to the X-ray beam, the spots will be symmetrically located on the pattern. In any other position no center of symmetry will exist. The other type of pattern is produced by a monochromatic beam impinging on a sample of a powdered crystalline material. This diagram consists of concentric rings corresponding to the diffraction interferences of the planes in the crystals.

If a crystal of a metal is in a thoroughly unstrained condition, the Laue spots will have a sharp outline. The shape of these spots is dependent upon the type of beam definition and production. Schiebold (2) has described the different shapes of these spots and the conditions which will produce each type. In usual practice they are elliptical because the beam is slightly divergent.

When the crystal is in a strained condition the Laue spots have an indefinite outline and tend to be elongated. This elongation is not radial, but along a definite curve. Berg (3) states that the curve of this striation is the locus of an undistorted Laue spot produced when the crystal is rotated about a crystallographic axis.

Schiebold and Sachs (4) have shown that the orientation of a crystal can be determined by making the stereographic projection of

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the unsymmetrical Laue diagram, filling in the great circles, and then, by the use of a stereographic net, rotate the projection until the great circle containing the cube faces, in the case of a cubic crystal, lies on the reference circle. The angle of rotation of the stereographic net is the angle of inclination of the crystallographic axes to the axis of the specimen as placed in the X-ray beam.

A simpler method of finding the orientation of a face-centered cubic crystal has been presented by Majima and Togino (5). They set up a single crystal of aluminum and made Laue diagrams for each five degrees of rotation of the specimen about a vertical and a horizontal axis. They (6) did a similar work on a single crystal of iron which has a body-centered cubic lattice. A comparison of an unsymmetrical Laue diagram with this series will locate the orientation of the specimen within less than five degrees.

These methods of determining the orientation of a crystal have been useful in the study of the mechanism of distortion. Carpenter, Elam, Wilson, and Taylor (7) used stereographic projection of crystals of aluminum where the external faces were indexed by optical means. The changes of these faces were followed by means of lines on the surface. The changes were then added to the original stereographic projections. In this manner the movement of the face pole of a plane could be followed. They conclude that slip takes place along the (111) family of planes and the direction of slip is toward the face pole of the (110) plane. The slip takes place first on one octahedral, (111), plane and when a definite amount of slip has resulted, additional slip then occurs on another (111) plane.

The use of X-rays in studying the mechanism of deformation of aluminum crystals was first reported by Taylor (8). He stated that crystal fragments had formed on the slip planes and could be compared to rollers between the planes. His experiments showed further that the crystal particles had rotated in one direction transverse to the direction of slip.

Yamaguchi (9) showed that the amount of rotation of these fragments increased with the amount of shear for each crystal and was greater for hard crystals than soft.

Burgers (10) investigated the orientation of the crystals produced by annealing the worked specimen. A comparison of this orientation with that in the worked unannealed crystal showed that the crystal fragments had rotated as much as sixty degrees in some instances, during deformation.

Gough (11) prepared a single crystal of aluminum and subjected it to deformation. He then sectioned the crystal and determined intensities of reflections from different planes. This procedure led him to believe that the crystal break-up during deformation formed small crystals which are oriented by only small rotations, about two degrees, about the axis contained by the slip plane and perpendicular to the slip direction.

The amount of rotation of these crystal fragments has been estimated from the elongated Laue spots to be about ten to twenty degrees. It is conceivable that this limitation could be imposed by the fact that a limited range of wave lengths of sufficient intensity is present in the beam. This would reduce the possible length of the striations, which, in turn, would limit the apparent range of rotation of the crystal fragments.

Joffe (12) made Laue diagrams of a bent crystal of rock salt. In the distorted patterns he found one spot which remained unchanged. This led him to the conclusion that a displacement and rotation of the broken parts of the crystal corresponded to a slip in a definite crystallographic plane and rotation around an axis perpendicular to the plane which yielded the unchanged spot. In a similar manner, Komar (13) made theoretical calculations as to the size and shape of the diffraction interferences from bent sodium chloride crystals and found actual photograms in good agreement with the calculated ones. However, he did not take into account the effect of any of the following limitations on the experimental method: the voltage on the tube, the true divergence of the beam, the fragmentation of the sample, or the time of exposure. All of these factors will profoundly affect the size and shape of any distorted Laue spot. These two investigators are the only ones who have reported obtaining a distorted X-ray pattern in which some of the spots have remained unchanged.

The first calculation of the lengthening of Laue spots was made by Rinne (14). A number of other investigators made similar calculations and Berg (3), using a sodium chloride crystal and light instead of X-rays, made complete calculations as to the shape of the elongated spots. He contended that the deformation can be assumed to be the result of bending about three axes. One of the axes is parallel to the X-ray beam and the other two perpendicular to it. Bending of a family of planes about the axis coincident with the primary beam will elongate the spot tangentially with respect to the

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center of the pattern. Bending about the other axes will produce elongation in a radial direction. In a transmission pattern the effect of bending about the primary beam will be negligible, therefore, the spots tend only to elongate in a nearly radial direction. In a reflected pattern the diffraction takes place from the layers on the surface and the spots are smeared in all directions.

A combination of the above theories of the mechanism of deformation would appear to more nearly explain the actual facts found during the deformation of a metal. This theory might be stated as follows: The first step in deformation is a bending of the crystal about at least two axes, followed then by a fragmentation along a set of planes. With additional stress, producing further deformation, the bending and fragmentation cycle again take place. Coincident with this second step is the rotation and translation of the previously formed fragments. This takes place repeatedly until fracture occurs. This idea is somewhat similar to the "step-wise slip" theory of Joffe (12) although no actual slip along planes takes place.

This mechanism of deformation would give rise to the slip lines seen under the microscope. These regions along crystal planes, where separation and fragmentation has occurred, would appear as straight lines using relatively low magnification. Under conditions of bending these would be expected to appear as curved lines. This is the case when viewed under high magnification.

If parallel slip took place in a crystal, the Laue diagram obtained after slip would have the same structure as before. The only change possible would be a small amount of striation due to the fragments formed, if Taylor's (8) theory is correct. The intensity of the striated portion of the Laue spot would be very much less than that for the spot caused by diffraction from the plane. The striation is considered by both Taylor and Yamaguchi to be caused by these fragments. In the actual patterns this change in intensity is not observed. The distribution of intensity appears to be the same as that found for the primary beam, at least in a qualitative measure. Wyckoff (15) showed the intensity distribution of the radiation from a number of targets at a fixed applied voltage. He referred to the original papers of Ulrey (16) and Kulenkampff (17) where additional curves of the same type can be found.

This uniform variation of intensity throughout the elongated spot is the result of a change in the large crystal itself. We have seen that parallel slip would have no effect. Therefore, the only alter-

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native, which would support the facts, is that the crystal has separated, and a small displacement of the separate parts has taken place. This would allow each set of planes of the crystal parts to select a wider range of X-rays for diffraction. The result of this wider selection would be an elongated spot with an intensity distribution similar to that of the primary beam.

It has been found that, when crystals are deformed, there is little or no change in the relative spacings of the planes in the crystal. This imposes a limitation on the theory of bending, namely, that this bending can only be over a small range. In other words, the radius of curvature of the plane which is bent must be large. If a cube is bent about a face, the perpendicular distance between any two parallel planes, which are perpendicular to the base of the cube, will be decreased. In a similar manner, the distance between any two parallel planes will be changed. Since only a slight change is found, then, fragmentation must take place after slight bending. This mechanism permits a displacement of one section of a crystal with respect to another. This will cause a change in the Laue spots.

The general theories of deformation have been applied to a polycrystalline material. Elam (7) has summarized the effects of mechanical deformation of the structure and properties of this type of material as follows:

- 1. The individual grains become distorted.
- 2. The deformation takes place by gliding on the crystal planes.
- 3. The presence of crystal boundaries strengthens the material.
- 4. The crystals tend to take up certain definite orientations in relation to the deformation.
- 5. The type of crystal lattice determines the orientation taken up.
- 6. The mechanical properties are changed by plastic deformation.
- 7. There is a real hardening of the material, i.e., deformation tends to increase the resistance to further deformation.
- 8. Change in orientation can only account for part of the effects of mechanical deformation.

The same concept as presented above for single crystals, that of bending followed by fragmentation, is probably the mechanism of deformation of polycrystalline material. Beckwith and Clark (18) have found that the deformation of silicon steel by rolling resulted in a periodicity of the phenomenon of fragmentation. Under the conditions used this occurred approximately at each 3.3 per cent reduction in area.

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(18) ted in condiaction The above mechanism of distortion is a combination of the theories of Joffe, Taylor, Yamaguchi. A similar idea was suggested by Mark, Polanyi, and Schmid (19), wherein they contend that the planes of the crystal are broken up into a system of blocks which are nearly parallel but each block itself being free from distortion. Polanyi contends that this bending and block formation is purely elastic and that recovery will take place when the deforming load is

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removed. It is evident that this cannot be the case.

A study was made of both single crystals of aluminum and of samples of polycrystalline aluminum. The obtaining of the deformation by tension was chosen because the form in which large single crystals are most easily obtainable is from thin sheet material.

Single crystals can be prepared in a number of ways. Elam (7) lists the following methods:

- 1. Growth from the molten bath.
- 2. Growth in the solid state.
- 3. Deposition of the crystal from the vapor phase.

The second method was chosen because of its simplicity of procedure and adaptability to aluminum. Further, the specimen could be machined prior to treatment and no effects of the machining would be found in the prepared sample. The procedure followed was that of Carpenter and Elam (20). The machined samples of aluminum,

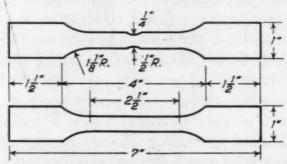


Fig. 1—Diagram of Test Bar for Thin sheet Aluminum Specimens. Thickness .018 to .025 Inch.

the dimensions and shape of which are shown in Fig. 1, were annealed in an electric muffle furnace for about forty-eight hours at a temperature of 400 degrees Cent. (750 degrees Fahr.). They were allowed to cool slowly to room temperature requiring a period of twenty-four hours. These samples were then given a permanent set of approxi-

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mately 2 per cent. This step is the most critical one of the procedure. Any set greater or less than this will not result in very large crystals. Following this strain treatment, the specimen was again annealed in an electric muffle furnace. This second anneal was started at a temperature of 300 degrees Cent. (570 degrees Fahr.), and the temperature gradually raised, over a period of four days, until the temperature reached 500 degrees Cent. (930 degrees Fahr.). The temperature was then raised to 560 degrees Cent. (1030 degrees Fahr.), for one hour. This was done to allow the small crystals on the surface to be absorbed. The final step then was to lower the temperature slowly for a period of two days until the sample had returned to room temperature. The chance of success in forming a single crystal in the center portion of the sample was about one in twenty. In all cases, however, there were usually not more than four or five crystals contained in the entire narrow section of the specimen. The second type of specimen shown in Fig. 1 had the stress concentration notch machined in the sample after the strain treatment and before the final annealing process.

These specimens were made of commercially pure aluminum sheet, about 0.018 inch thick, and machined to the dimensions shown in Fig. 1. This thickness permitted taking the X-ray patterns in approximately fifteen minutes.

It was desired to follow one crystal in each specimen throughout the deformation from the annealed state to fracture. Any previous work done in this field followed the procedure of taking a picture, deforming the piece in some manner, remounting the specimen on the X-ray tube, and taking a pattern to show the condition after distortion. This method did not insure that the same portion of the specimen was in the field of the beam when each picture was taken.

In order to be sure that all successive patterns showed the condition of the same part of the specimen, a special tension testing machine was designed and built. This machine is shown in Fig. 2. It is small enough to fit on the table of the X-ray tube assembly and to be placed between the defining pinhole system and the film. The grips, which held the specimen, were tapered and cross-hatched on the specimen side to prevent slipping. The loading was accomplished by a screw head that operated through a thrust bearing on a spring. The springs used were calibrated in the machine by means of standard weights placed on a pan supported by the upper grip assembly. An Ames dial, reading directly to 0.01 millimeter, was attached to the

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frame of the machine and an arm was affixed to the plunger of the dial. This arm was displaced in a vertical direction by the movement of the screw head. This dial was used in both the calibration of the springs and the actual testing of the specimen.

An attempt was made in the design of this machine to minimize the possibility of eccentric loading. This type of loading will produce

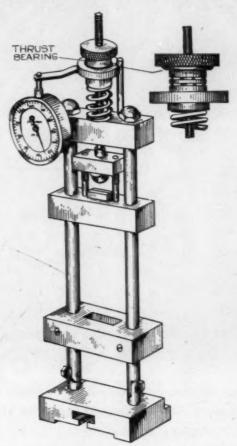


Fig. 2—Diagram of Device for Applying Tensile Stress to Thin Sheet Specimens.

bending in the specimen and will result in a tearing action at the point of fracture. This ideal condition was probably not fully obtained.

The orientation of the crystals, in the specimens used, was determined by comparison of the diagram obtained with the standard patterns reported by Majima and Togino (5). All the crystals produced by the strain-anneal method were found to have nearly the same orientation. The (100) face was inclined approximately 75 degrees from the horizontal plane containing the X-ray beam and had

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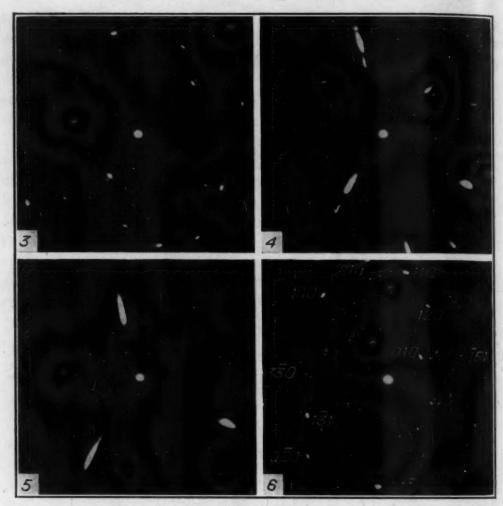


Fig. 3—X-ray Photogram of Specimen 3A. Stress = 0. Elongation = 0.
Fig. 4—X-ray Diffraction Pattern of Specimen 3A. Stress = 6000. Elongation = 4 Per Cent.
Fig. 5—X-ray Diffraction Pattern of Specimen 3A. Stress = Residual 0.5 cm. Below Fracture.
Fig. 6—X-ray Diffraction Pattern of Specimen 11A. Stress = 0. Elongation = 0.

an inclination of approximately 68 degrees from the vertical plane containing the X-ray beam.

Each series of pictures of a specimen were taken with it remaining in the same position in the beam throughout the entire test. The load was applied in steps and the exposures were made immediately after the application of the load, while the load was acting on the specimen.

A series of diffraction patterns of single crystals and representative ones from two other series are included herein. These are shown in Figs. 3 to 10 inclusive. One complete series and representative pictures from two other series of tests on polycrystalline aluminum are shown in Figs. 11 to 16 inclusive.

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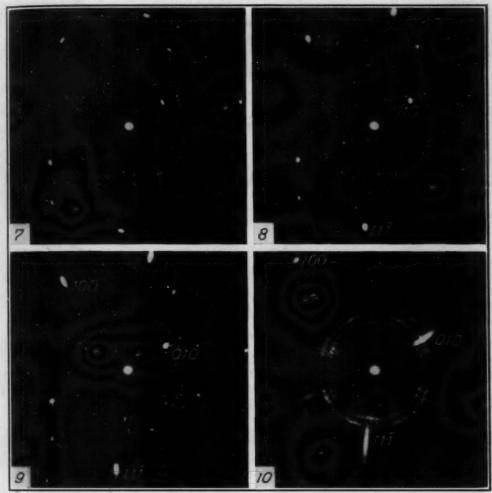


Fig. 7—X-ray Diffraction Pattern of Specimen 11A. Stress = 2000. Elongation = 0.5 Per Cent.
Fig. 8—X-ray Diffraction Pattern of Specimen 11A. Stress = 4000. Elongation = 1.5 Per Cent.
Fig. 9—X-ray Diffraction Pattern of Specimen 11A. Stress = 5000. Elongation = 2.3 Per Cent.
Fig. 10—X-ray Diffraction Pattern of Specimen 11A. Stress = 10,900. Elongation = 20.9 Per Cent.

The conditions under which each X-ray diagram referred to above are shown in Table I and II.

The remaining two pictures not listed in the tables were made on a specimen of the type which had a stress concentration notch machined in the narrow section of the test piece. This resulted in the fracture occurring at the point where the X-ray beam passed through the sample.

The sheet from which these samples were prepared showed some preferred orientation as a result of rolling. This sheet was annealed and sample number 23A was cut so as to have the direction of rolling

Table I
Single Crystal Samples

		re 15 minutes at 30 K.V. ar given as per cent in 6 cm.	
Fig. No.	Experiment No.	Stress Acting lbs. per sq. in.	Comments
3 .	.3A1 .3A9	0 6000	Original 4 per cent
5	3A11	residual stress residual stress	elongation after fracture at point of fracture
	Specimen 3A broke at beam used in Figs. (3-	a point about 0.5 cm. above -5).	the location of the
6 7	11A1 11A2	-0 2000	Original 0.5 per cent elongation
	Load removed for 5 0.16 per cent of the e	minutes and the specimen longation.	recovered all but
			0.7 per cent elongation
	Load removed and the	specimen did not recover.	
8	11A5	4000	1.5 per cent
9	11A6	5000	elongation 2.3 per cent
10	11A12	Dial removed to prevent damage by fracture.	elongation 20.9 per cent elongation
	Specimen broke about	1.5 cm. below beam.	

Table II
Polycrystalline Aluminum Specimen

	30 minute ex	eposure at 30 K.V. as	nd 25 M.A.
Fig.	Experiment No.	Stress Acting lbs. per sq. in.	Comments
11 12	10A1 10A11	7000	Original 1.5 per cent elongation
	At this point the load wa heavy spring put in t	as released and the li	ght spring removed and
13	10A16	12000	10 per cent elongation
	The specimen continued 12500 could be applied.	to stretch but no l This same applies to	oad greater than about the next figure.
	The specimen broke 1.6 applied.	cm. above beam wh	nen additional load was
14	10A19	residual stres	after fracture

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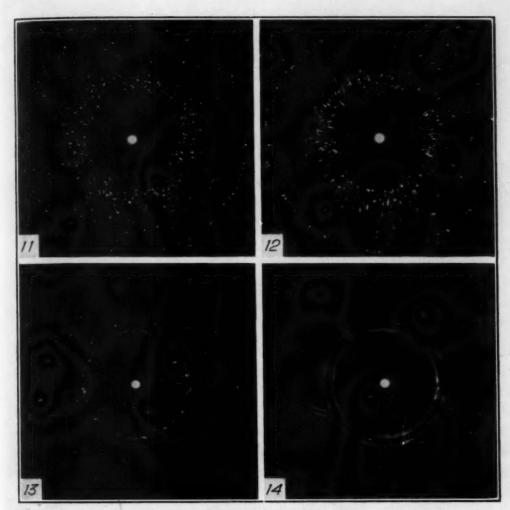


Fig. 11—X-ray Diffraction Pattern of Specimen 10A. Stress = 0. Elongation = 0. Fig. 12—X-ray Diffraction Pattern of Specimen. Stressed 7000, Elongation 1.5 Per Cent. Fig. 13—X-ray Diffraction Pattern of Specimen. Stressed 12,000, Elongation 10 Per Cent. Fig. 14—X-ray Diffraction Pattern of Specimen with Residual Stress 1.6 cm. Below Fracture.

of the sheet the axis of the specimen. Sample number 24A was cut from the same sheet with the axis of the specimen inclined 15 degrees to the direction of rolling of the sheet.

Fig. 15 is an X-ray picture of the original condition of sample 23A. The intensity maxima can be seen on the spotted rings. Fig. 16 is an X-ray picture showing the result of an applied load which caused fracture.

RESULTS

The series of patterns obtained from the tests made on sample number 11A will be considered first. These are represented by Figs. 6 to 10 inclusive. The first change in the Laue spots is a shadow

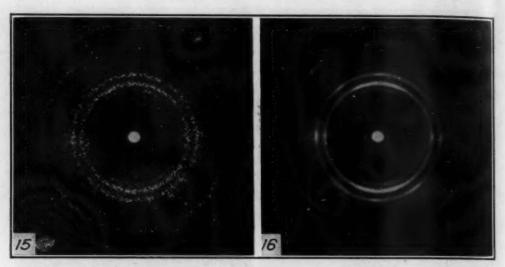


Fig. 15—X-ray Diffraction Pattern of Specimen 24A (notched), original state. Fig. 16—X-ray Diffraction Pattern of Specimen 24A after fracture, showing preferred orientation.

appearing below the interference from the (111) plane. The position on the film of this shadow corresponds to the Ka interference from the 222 planes of a powder specimen. This interference can be seen throughout the entire series as a maximum intensity point on the striation of the Laue spot from the (111) plane. Similar intensity maxima can be seen along the other striations. Wherever the striation crosses a point on the film corresponding to an interference position of a family of planes which are of some order of the diffraction spot from the single crystal, these maxima will appear. This is a result of the fact that the intensities of the $K\beta$ or $K\alpha$ wave lengths in the radiation are much greater than that of the white radiation background. If the striation of a spot from a single plane does not cross a position occupied by a diffraction ring or some family of planes, only of higher or lower order, no maxima will appear. These two facts are true only below the stage of fragmentation where the amount of fragmented crystalline material in the X-ray beam is less than that of the single crystal. When this stage in the deformation is exceeded, then maxima will occur along all the striations wherever a diffraction ring position is crossed. This can be seen in Fig. 10 on the (111) striation. Finally, continued fragmentation and rotation of these fragments of the crystal cause the reduction in intensity of the striation and the appearance of maxima corresponding to preferred orientation. The continual change in the Laue spot from the (100) face shows this effect clearly. In Fig. 10 the intensity 1937

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maximum corresponds to the position of the Ka interference from the (400) planes, the fourth order of the (100) planes.

In any of the diagrams which show elongated spots, there are two sharp edges on these striations. The one farther from the center corresponds to the bromine absorption edge of an interference which the striation crosses. The one nearer the center corresponds to the silver absorption edge for the same interference.

Figs. 3 to 5, representing samples 3A and 12A, show the same characteristics and follow the same stages of deformation.

The mechanism of deformation proposed in the theoretical part of this section demands that the intensity distribution along a striation follow the theoretical intensity curve for the primary beam. This is found to be the case. As the intensity of a striation decreases, maxima appear which are caused by diffraction of the Ka or $K\beta$ radiation by small crystals in a preferred orientation.

Therefore, these facts tend to support the theory that the distortion, in a single crystal, is bending followed by fragmentation with

subsequent preferred orientation of the crystal fragments.

The series of X-ray diffraction patterns of polycrystalline aluminum, represented by Figs. 11 to 16 inclusive, show that the mechanism of distortion is the same for an aggregate of small crystals as for a single crystal. The change in each small spot is the same as the change for the identical spot from a single crystal. This experiment further proves the theory because it shows that the final stage in deformation is fragmentation into a very large number of small crystals. This is shown by the production of smooth Debye-Scherrer rings. The rotation of the fragmented crystals is shown by the formation of intensity maxima on the rings. This is an indication of preferre orientation.

The amount of preferred orientation in sample 23A was increased by the action of the tensile load. This is shown by the increase of the intensity of the maxima shown on the rings in Fig. 16 over that already present on the rings shown in Fig. 15.

The action of the tensile load on specimen number 24A changed the direction of preferred orientation. This is shown by the fact that the intensity maxima in the original are situated at an angle of about 15 degrees from the vertical and horizontal axes of the pattern, whereas the intensity maxima in the pattern obtained after fracture are situated at an angle of about 10 degrees from the axes of the diagram.

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This indicates that not only is preferred orientation a result of deformation, but, that during deformation, an existing preferred orientation in a different direction will be changed into the type produced by the particular kind of mechanical working employed. This is further proof of the theory presented in that it illustrates the rotation and translation of small crystal fragments.

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DISCUSSION

Written Discussion: By N. P. Goss, Cold Metal Process Co., Youngstown, Ohio.

The interpretation of radial asterism and appearance of Debye-Scherrer rings when deformed metals are X-rayed as presented by the authors is not new. It is, however, gratifying to find that Professor Clark has abandoned his former interpretation of "radial asterism" as defined in his book1 and other publications. A brief review of the former interpretation will be discussed so that one who is not versed in this subject will be so advised. Professor Clark's former interpretation was erroneous, and similar to that proposed by Czrochralski. Elam² has

¹G. L. Clark, "Applied X-rays," McGraw-Hill Book Co., New York City. ²C. F. Elam, "Distortion of Metal Crystals," Oxford, 1935, p. 169.

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also discussed this in her book, to quote, "The elongation of the Laue spots first described by Czrochralski was also interpreted by him as indicating a gradual distortion of the lattice, involving alteration in the spacing of the planes. Photographs of severely worked metals show that this is not true, the spacing of a particular substance remaining constant except for a small distortion within the elastic range. Evidence of a still greater amount of breaking up is obtained from the fact that the reflected spots from a perfect crystal are replaced by Debye-Scherrer rings when the crystal is severely worked."

On page 1221 the authors say that: "This experiment further proves the theory because it shows that the final stage in deformation is fragmentation into a large number of small crystals. This is shown by the production of smooth Debye-Scherrer rings." This is the same as the interpretation given by Elam, and by Jeffries and Archer.

The same experiment as described in this paper was performed by Jeffries and Bain, and the results were published in 1921. They found by X-ray analysis that the first mechanical working of coarse-grained metals produced a mixed orientation of the crystal fragments within the large grains, and that cold working produces a structure which simulates in many respects a very fine-grained metal. A search of the literature proves that Bain and Jeffries were the first to prove that deforming a single grain produced a mixed orientation of smaller crystal fragments.

I would like to ask Professor Clark whether he has abandoned his earlier interpretation of "radial asterism" in favor of the one indicated here and which is in substantial agreement with that given in several of my own papers.

In 1928 I studied single crystals of metals and other substances with the X-ray, and found that many of the single crystals exhibited "asterism" (elongated diffraction spots). In some instances the elongated spots consisted of a number of parallel striations (clearly resolved). When filtered X-ray radiation was used, the radial streaks disappeared and only a single sharp spot remained [(due to K^{α} or K^{β}) radiation]. Experimenting along these lines I found these crystals to be built up of smaller units. Such structures are now called mosaics. These researches convinced me to consider "asterism" to be associated with the "fine structure" of the grains, and not lattice distortion. However, more investigators thought that "asterism," etc., meant lattice distortion and so little attention was paid to my work and that of others. However, the viewpoint is gradually changing to the one I gave several years ago. When accepted universally, the X-ray will then be of the utmost value in studying the internal structure of the grains. The possibilities were described in the December 1936 issue of Transactions and the discussions by Goetz and Cramer are of unusual interest.

³Jeffries and Archer, "Science of Metals," McGraw-Hill Book Co., New York City, p. 65, p. 205-209 (cause of strain hardening).

⁴Mixed Orientations Developed in Crystals of Ductile Metals by Plastic Deformation, Chemical and Metallurgical Engineering, Vol. 25, p. 775, Oct. 26, 1921.

⁵Transactions, American Society for Metals, Dec. 1936, p. 977; p. 1029 (discussion by Nusbaum).

TRANSACTIONS, American Society for Metals, Dec. 1936, p. 977; p. 1029 (discussion by Nusbaum).

TRANSACTIONS, American Society for Metals, December 1936, p. 1030, discussion by Goetz; p. 1024, discussion by Cramer.

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The authors show that the final stage of deformation is fragmentation, but I fail to see how the X-ray diagrams show that the initial stage is one of bending. What is bent, and how do the X-ray diagrams show this?

On page 1208 the authors make the statement that the elliptical spots are due to the divergence of the X-ray beam; however, I believe they meant that this is due to the angle at which the pinhole system intercepts the X-ray beam from the target, i.e., at an angle of about 5 degrees, and this would make the focal spot appear elliptical.

Authors' Reply to Discussion

Since the presentation of this paper, some of the questions raised by Mr. Goss have been subjected to very critical study. The results are presented in a paper by G. L. Clark and C. G. Dunn, entitled "X-Ray and Magnetic Analyses of Deformed Silicon Steel Rings." This paper presents an analysis of greatly enlarged patterns made with carefully filtered radiation essentially monochromatic.

Flat rings cut from sheet silicon by grinding and etching, so as best to preserve the properties of uncut sheet, were clamped between dies that bent them to fit more or less closely over a toroidal plunger, and allowed, under these conditions, testing of circumferential magnetization and taking of X-ray diffraction patterns (Laue type) at one point on the most deformed circle.

Slight deformation temporarily shifts and elongates the Laue spots and depresses the magnetization curve. More severe deformation permanently displaces some Laue spots, though the crystals in their new orientations are not permanently strained. The magnetization curve is permanently depressed, and it is suggested that this is due to changes at grain boundaries not recorded by the X-ray method. Very severe deformation leaves some crystals strained, as shown by X-rays, and the permanent changes in magnetic properties are nearly as great as the temporary changes. The changes in shape of Laue spots in coarse-grained rings agree at all stages with bending of strained crystals about axes lying in the original plane of the sheet.

Specimens cut from partly annealed sheets that had previously been given various reductions in thickness by cold rolling showed parallel but rather erratic changes in magnetic hardness, measured by inverse maximum permeability, and X-ray strains, measured by range in angles of incidence on single crystals.

⁸Physical Review, Vol. 52 (in press).

TECHNICAL PROGRAM AND REPORTS OF OFFICERS AMERICAN SOCIETY FOR METALS—19th ANNUAL CONVENTION, ATLANTIC CITY, OCTOBER 18 to 22, 1937

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OR purposes of record and for the benefit of members who were not in attendance at the Nineteenth Annual Convention of the Society, held in Atlantic City, October 18 to 22, 1937, the Technical Papers Program and Reports of Officers presented at the annual meeting are herewith published in full.

MONDAY, OCTOBER 18

Morning Session-Ambassador Hotel-Renaissance Room

Joint Chairmen—G. B. Waterhouse and H. J. French The Process of Dry Cyaniding, R. J. Cowan and J. T. Bryce, Surface Combustion Corp., Toledo, Ohio.

The Mechanism of Steel Hardening and Tempering as Indicated by Coercive Force Measurements, R. S. Dean, Bureau of Mines, Washington, and C. Y.

Clayton, Missouri School of Mines and Metallurgy, Rolla, Mo. Effect of Tempering Quenched Hypereutectoid Steels on the Physical Properties and Microstructure, C. R. Austin and B. S. Norris, Pennsylvania State College, State College, Pa.

Afternoon Session-Auditorium Ball Room

Joint Chairmen-J. P. Gill and O. V. Greene

Effect of Carbon on the Hardenability of High Purity Iron-Carbon Alloys, T. G. Digges, National Bureau of Standards, Washington.

Precipitation-Hardening of Cobalt Steel: A New Tool Material, R. H. Har-

rington, General Electric Co., Schenectady, N. Y.
Quantitative Hardenability, J. L. Burns, T. L. Moore and R. S. Archer, Re-

public Steel Corp., Chicago.

Afternoon Simultaneous Session-Auditorium-Room B

Joint Chairmen-R. L. Dowdell and N. L. Mochel

A Note on Rapid Photomicrography, W. Mutchler and H. O. Willier, National

Bureau of Standards, Washington. The Effect of Grain Size on the Oxidation of a Low Carbon Steel, C. A. Siebert and Clair Upthegrove, University of Michigan, Ann Arbor, Mich.

Electromagnetic Measurements and Steel Structures Correlated, Carl Kinsley, Consulting Engineer, New York City.

Auditorium Ball Room

Educational Lectures

Open-Hearth Steel Making, Earnshaw Cook, American Brake Shoe & Foundry Co., Mahwah, N. J. Metallographic Technique, J. R. Vilella, United States Steel Corp., Kearny, N. J.

TUESDAY, OCTOBER 19

Morning Session-Ambassador Hotel-Renaissance Room

Joint Chairmen-A. A. Bates and V. O. Homerberg
The Rupture Strength of Steels at Elevated Temperatures, A. E. White and C. L. Clark, University of Michigan, Ann Arbor, Mich., and R. L. Wilson, Timken Roller Bearing Co., Canton, Ohio.

The Electrode Salt Bath for Hardening High Speed Steel, Axel Hultgren,

Consulting Metallurgist, Stockholm, Sweden.

A New Application for the Short-Time High Temperature Tensile Test, C.
L. Clark, A. E. White and G. J. Guarnieri, University of Michigan, Ann Arbor, Mich.

Afternoon Session-Auditorium Ball Room

Joint Chairmen-G. R. Brophy and R. F. Mehl

Austenitic Grain Size of Eutectoid Steel, Harry Tobin and R. L. Kenyon, American Rolling Mill Co., Middletown, Ohio.

Some Factors Influencing Austenitic Grain Size in High Purity Steels, G. Derge, Carnegie Institute of Technology, A. R. Kommel, United Engineering and Foundry Co., Vandergrift, Pa., and R. F. Mehl, Carnegie Institute of Technology, Pittsburgh.

Relation of Pre-Treatment of Steele to Austenitic Grain Greath L. E. D.

Relation of Pre-Treatment of Steels to Austenitic Grain Growth, J. E. Dorn and O. E. Harder, Battelle Memorial Institute, Columbus, Ohio.

Auditorium Ball Room

Educational Lectures

Open-Hearth Steel Making, Earnshaw Cook, American Brake Shoe & Foundry Co., Mahwah, N. J.

Metallographic Technique, J. R. Vilella, United States Steel Corp., Kearny, N. J.

WEDNESDAY, OCTOBER 20

Morning Session-Ambassador Hotel-Renaissance Room

ANNUAL MEETING OF THE A.S.M.

1937 Edward De Mille Campbell Memorial Lecture presented by W. P. Sykes of the General Electric Co., Cleveland, entitled "Structural and Hardening Characteristics of Some Iron-Cobalt-Tungsten Alloys" Chairman-Prof. H. M. Boylston

Afternoon Session—Auditorium Ball Room

Joint Chairmen—H. W. McQuaid and N. I. Stotz
The Effect of Mass Upon the Mechanical Properties of Cast Steel, C. W.

Briggs and R. A. Gezelius, U. S. Naval Research Laboratories, Washington. The Fatigue Resistance of Steel as Affected by Some Cleaning Methods, J. H. Frye, Jr., and G. L. Kehl, LeHigh University, Bethlehem, Pa. Influence of Heat Treatment on Creep of Carbon-Molybdenum and Chromium-Molybdenum-Silicon Steel, R. F. Miller, R. F. Campbell, R. H. Aborn, U. S. Steel Corp., Kearny, N. J., and E. C. Wright, National Tube Co., Pitteburgh Pittsburgh.

Auditorium Ball Room

Educational Lectures

Open-Hearth Steel Making, Earnshaw Cook, American Brake Shoe & Foundry Co., Mahwah, N. J.

Metallographic Technique, J. R. Vilella, United States Steel Corp., Kearny, N. J.

THURSDAY, OCTOBER 21

Morning Session-Ambassador Hotel-Renaissance Room

Joint Chairmen-O. E. Harder and J. F. Harper

Symposium on Carburizing

A Review of Some Fundamentals of Carburizing, M. A. Grossmann, Carnegie-Illinois Steel Corp., Chicago.

A Theoretical Discussion of the Action of Solid Carburizing Agents, H. W. McQuaid, Republic Steel Corp., Massillon, Ohio.

Gaseous Media for Carburizing, G. T. Williams, Cleveland Tractor Co.,

Cleveland.

Morning Simultaneous Session-Ambassador Hotel-Rainbow Room

Joint Chairmen-Howard Handy and Howard Scott

The Initial Stages of Graphitisation, H. A. Schwartz and M. K. Barnett,

National Malleable and Steel Castings Co., Cleveland.

An X-Ray Study of the Ars and Acs Points of Iron and Iron-Nickel Alloys, Sidney D. Smith, Pennsylvania State College, State College, Pa.

Graphitization in High Purity Iron-Carbon Alloys, Cyril Wells, Carnegie

Institute of Technology, Pittsburgh.

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Afternoon Session-Auditorium Ball Room

Joint Chairmen-R. G. Guthrie and John Wyzalek

Symposium on Carburizing

Factors Governing Selection of Type of Carburized Case, A. L. Boegehold and C. J. Tobin, General Motors Corp., Detroit.

Furnace Atmospheres and Decarburisation, J. A. Webber, Interstate Drop Forge Co., Milwaukee.

Steels Used in the Carburizing Process, O. W. McMullan, Youngstown Sheet

& Tube Co., E. Chicago, Ind.

A Hardenability Test for Carburizing Steel, W. E. Jominy and A. L. Boegehold, General Motors Corp., Detroit.

Afternoon Simultaneous Session-Auditorium-Room B

Joint Chairmen-H. E. Brown and S. L. Hoyt

Solubility of Copper in the Grain-Boundary Material of a Solid Solution of Copper in Zinc, G. R. Dean, Miner Laboratories, Chicago, and W. P. Davey, Pennsylvania State College, State College, Pa.

The Rate of Austenite Transformation in Cast Iron, D. W. Murphy, W. P.

Wood and D. Girardi, University of Michigan, Ann Arbor, Mich.

The Effects of Columbium and Other Addition Agents on Low Chromium Steels, Russell Franks, Union Carbide and Carbon Research Laboratories, Inc., Niagara Falls, N. Y.

Auditorium Ball Room

Educational Lecture

Open-Hearth Steel Making, Earnshaw Cook, American Brake Shoe & Foundry Co., Mahwah, N. J.

FRIDAY, OCTOBER 22

Morning Session-Auditorium Ball Room

Joint Chairmen-R. S. Archer and A. A. Bates

Symposium on Carburizing

Production Carburising, E. F. Davis, Warner Gear Div., Borg-Warner Corp., Muncie, Ind.

A Study of Commercial Carburizing Containers, R. W. Roush and A. C.

Dames, Timken-Detroit Axle Co., Detroit.

The Physical and Chemical Characteristics of Carburizing Compounds and Their Handling in Production, S. L. Widrig, Spicer Manufacturing Co., Toledo, Ohio.

Morning Simultaneous Session—Auditorium—Room B

Joint Chairmen-Bradley Stoughton and K. R. VanHorn

Effects of Longitudinal Scratches on Valve Spring Wire, F. P. Zimmerli, Barnes-Gibson-Raymond, Detroit, W. P. Wood, University of Michigan, Ann Arbor, Mich., and G. D. Wilson, Barnes-Gibson-Raymond, Detroit. Recovery of Cold-Worked Nickel on Annealing, Erich Fetz, Wilbur B. Driver Co., Newark, N. J.

Metal Coloring, C. B. F. Young, Columbia University, New York City.

Afternoon Session—Auditorium Ball Room

Joint Chairmen-M. A. Grossmann and L. A. Lanning

Symposium on Carburizing

Commercial Gas Carburising, L. D. Gable and E. S. Rowland, Timken Roller Bearing Co., Canton, Ohio.

Light Cases by Gas Carburizing, V. T. Malcolm, Chapman Valve Manufac-

turing Co., Indian Orchard, Mass.

Liquid Bath Carburizing, B. B. Beckwith, Chrysler Corp., Detroit.

Afternoon Simultaneous Session-Auditorium-Room B

Joint Chairmen—T. H. Nelson and E. F. Ross

A Study of Deoxidation Type Inclusions in Alloy Steels, W. A. Hare and
Gilbert Soler, Timken Roller Bearing Co., Canton, Ohio.

Some Problems in the Production of Low-Carbon Sheets in Non-Continuous

Some Problems in the Production of Low-Carbon Sheets in Non-Continuous Mills, M. L. Samuels and Alfred Boyles, Battelle Memorial Institute, Columbus, Ohio.

Relation of Size of Spheroids in Tool Steel to its Machinability and to Holding Edge of Cutter, D. E. Roda, International Business Machines Corp., Rochester, N. Y.

Auditorium Ball Room

Educational Lecture

Open-Hearth Steel Making, Earnshaw Cook, American Brake Shoe & Foundry
Co., Mahwah, N. J.

ANNUAL ADDRESS OF THE PRESIDENT

Nineteenth Annual Convention, Atlantic City, October 20, 1937 EDGAR C. BAIN, President

THE Society has fared well, and has enjoyed an exceedingly successful year of growth and great vitality during the term of office of your retiring President; but this good fortune is not to be attributed to him. Rather the usefulness and vigor of our Society result from the enthusiasm of its members and the willingness to work of those elected as Chapter Officers. During the year augmented services have been rendered and the treasury is by no means depleted thereby, indeed we may reasonably expect that the functions of the Society may continue to be performed unless prolonged adversity should intervene.

Your President welcomes this opportunity to acknowledge before the membership his debt of gratitude to the Trustees of the Society for serious and whole-hearted interest in their Trust. It is right that you know that they have approached the problems confronting them without prejudice and so studiously that in essentially all cases, deliberation continued until unanimity of opinion was reached.

The Board of Trustees held four meetings during the year: November 20, 1936; April 30, 1937; August 20, 1937 and October 19, 1937, the first three at Cleveland, the latest at Atlantic City.

The President's Medal was presented by Past President Archer to Mr. B. F. Shepherd at the Annual Banquet in Cleveland, October 20, 1936. The Howe Medal was not awarded at that time due to a change in the rules of award authorized by the Board of Trustees to

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provide more time for consideration of papers after publication. Without omitting any papers from consideration the Committee has this year selected the most meritorious from those published during September and December, 1935, and March and June, 1936, and the award, based upon this selection, will be made at the banquet tomorrow night, October 21, 1937.

The splendid 1936 Campbell Memorial Lecture was presented by Mr. J. P. Gill on the subject, "High Speed Steel, Carbide Segregate and Grain Size." The Sauveur Achievement Award was made to Mr. W. D. Chapin at the Annual Banquet and a most gracious response from Mr. Chapin is recorded in the December, 1936, issue of Transactions. No award will be made this year due to a change in the Rules of Award providing a new mode of deliberation for the Committee of Past Presidents.

The Board of Trustees authorized the Secretary to extend an invitation to the British Iron and Steel Institute and the Institute of Metals to attend the National Congress in Detroit in 1938 when these Societies of Great Britain hold their meetings in America. The invitation has been accepted subject to the exigencies of their own meetings.

The Handbook Committee is already at work upon the production of a new Handbook, largely rewritten and revised. The present large edition is even now well along toward complete distribution and the new book may make its appearance in 1938. The Society is especially grateful to the many members who are keeping this fine book at the top in its class.

The Committee on Educational Lectures has arranged the two series of lectures being enthusiastically attended this week. In recognition of their excellence we take this opportunity of thanking Mr. Earnshaw Cook for his lectures on "Open-Hearth Steel Making," and Mr. J. R. Vilella for his evening lectures on "Metallographic Technique."

The Committee on Visual Education has made a wide inquiry into the most modern devices of presenting knowledge for rapid learning. Their investigations are already bearing fruit in listing available motion pictures and in the assembling of a film showing certain metallographic processes in a novel and illuminating way, most of which will probably be new to our members.

Complying with the express instructions of the Board of Trustees your President visited as many of the Chapters as was possible

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and attended the inauguration of three new Chapters—Puget Sound at Seattle, Oregon at Portland and Texas at Houston, it being impossible to attend the first meeting of the Calumet Chapter, embracing Hammond, Gary and East Chicago. These new Chapters were formed as a result of the enthusiastic interest of members and their friends in the various localities. Three new student groups were formed-at Virginia Polytechnic Institute, Michigan College of Mining and Technology and the Missouri School of Mines and Metallurgy.

Student interests are also fostered by a number of the other Chapters, such as Cleveland at Case School of Applied Science, Detroit at University of Michigan, Notre Dame at University of Notre Dame, Lehigh Valley at Lehigh University, Penn State at Penn State, Pittsburgh at Carnegie Institute, Peoria in the plant of the Caterpillar Tractor, Philadelphia at Temple University, and Boston at M.I.T. Another evidence of interest in addition to the new Chapters and groups operating under their own name is the extension work of the Detroit Chapter which, in addition to the group at the University of Michigan, also has active units under its protecting wings at Toledo, Lansing and Flint.

During the year the number of Chapters and groups has thus increased from 42 to 49 and the membership has crossed the 10,000 mark. By travelling some 23,000 miles the President was able to visit half of the Chapters, presenting a technical talk on all but two visits. Several Chapters had been visited during the previous year by the then Vice President. These visits provided an opportunity to observe the splendid work of the Chapters and particularly to discuss the educational courses. This aspect of Chapter activity is of special interest and may possibly be our outstanding service. It will, no doubt, be a surprise to many of you to learn that nearly 4000 men were enrolled in the classes sponsored last year by the 37 Chapters reporting, 26 of which held courses. About half of the courses were based upon the printed lectures available, with lantern slides, from the National Office. Further details will be published in the forthcoming issues of THE REVIEW.

The president takes this opportunity to acknowledge with sincere thanks the co-operation of the societies which have associated themselves with ours in this Congress: American Welding Society, The Wire Association, American Institute of Mining and Metallurgical Engineers.

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The President's Bell

Past President R. M. Bird in 1926 established the award of a bell (to call the faithful to Chapter sessions), to be made to Chapters of outstanding services. Not only for excellent educational work, not only for an inspiring sponsorship of studies at Temple University, and not wholly for the making available by publication of its exceedingly fine lectures on fundamentals—rather for a sustained quality of excellence, maintained at a mark set some 10 years ago, the President's Bell has been awarded to the Philadelphia Chapter. (Past President W. B. Coleman accepted the bell on behalf of the Philadelphia Chapter.)

In conclusion, a special call is made to urge all to co-operate in every way possible with the Handbook Committee and the Secretary, J. Edward Donnellan, as he invites your assistance with the Handbook that it may fairly represent the progressive attitude of the Society, and that it typify the Society's striving for technical accuracy.

It is my pleasant duty to acknowledge the splendid co-operation of the four technical societies—The American Welding Society; The Wire Association; and the Institute of Metals and Iron and Steel Divisions of the American Institute of Mining and Metallurgical Engineers—in extending the scope of the National Metal Congress and Exposition.

REPORT OF THE TREASURER

October 20, 1937

W. P. Woodside, Treasurer

The Society having completed on August 31, 1937, the most successful year to date, financially, in its history, it is the pleasure of the Treasurer to submit this report.

The cash position of the Society as shown on the audited statement has materially improved, the cash on hand and in the banks on August 31, 1936, being \$37,761.25 and on August 31, 1937, \$51,306.83, an increase of 30 per cent, while at the same time we have increased our security holdings by 20 per cent and our other assets by approximately 10 per cent. The only asset to decrease in total was our accounts receivable which indicates better collections and a healthier condition of our accounts. Inventories were in-

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creased by approximately \$2,300.00 entirely by additions to our paper and cover stock for our magazines. Our sales of books were quite heavy during the year and our inventory of books on hand was therefore considerably lower. Collection was affected on our restricted bank account which has eliminated this item.

The market value of our securities has maintained its close relation of last year with the amount invested or cost in spite of the late summer drop in the markets. The total amount invested is \$197,140.78. The market value on August 31, 1937, is \$181,416.61.

A reserve for market depreciation of \$25,000.00 is still carried to very amply take care of the difference.

Sales and calls of securities.

\$1000.00 American Gas and Electric Co. 5's 6000.00 Youngstown Sheet and Tube Co. 31/2's

Purchased.

\$5000.00 Pennsylvania R. R. Co. 4½'s 3000.00 Pennsylvania R. R. Co. 3¾'s 2000.00 Pennsylvania R. R. Co. 3¼'s

100 shares Massachusetts Investors Trust 100 shares Incorporated Investors 100 shares International Nickel Co. of Canada 100 shares Standard Oil Co. of New Jersey 100 shares Sears, Roebuck & Co.

Our portfolio now shows investment in

Government Bonds	\$54,000.00	28%
Utility Bonds	49,000.00	25%
Railroad Bonds	35,000.00	19%
Industrial Bonds	15,000.00	8%
Investment Trusts	16,500.00	9%
Stocks	21,500.00	11%

Most economists and trained business observers have been and still are prophesying inflation, and with rising commodity and labor markets it was felt that maintaining the number of dollars available to the Society through its investments was not sufficient but that an attempt should be at least partially made to preserve present dollar value which could be done by purchasing some of the highest grade stocks. These stocks will reflect more accurately and quickly inflation and rising prices as they occur.

Our audited statement of income and expense for the fiscal year ended August 31, 1937, is as follows in total:

Income Expense \$297,508.03 252,197.18

Net Gain

\$45,310.85

Advertising income from Metal Progress increased again this year by approximately 25 per cent over the prior year.

Total Income from METAL PROGRESS Total Expense from METAL PROGRESS \$105,640.68 96,204.21

Excess Income

\$9,436.47

Book sales again show a substantial contribution to our net profit and surplus for the year, the net amount of this year's activity being approximately \$7500.00.

The 1936 edition of the Metals Handbook was published and mailed to the members in September and October of this fiscal period and it is gratifying to find that the cost of this publication has very nearly been balanced in the first year by the income from advertising and sales.

The 1936 Cleveland Convention and Exposition in addition to being an outstanding success educationally and as an exhibition of beauty and sales value was also as usual a financial success.

The membership, which is our foundation for all activities, continues to grow and has reached new high totals in members and of course in receipts. We added approximately \$19,000.00 to our gross dues in the past period to make a total gross of \$105,600.00 and returned an additional \$7500.00 to the Chapters to make a total return of over \$40,000.00.

In addition to the figures shown on the audited reports we have reports from the Chapters showing a total of over \$58,000.00 in assets held by them. This figure is a further increase over the prior period and indicates a continued healthy financial position for the Chapters.

CONDENSED AUDITED BALANCE SHEET AMERICAN SOCIETY for METALS

As of August 31, 1937

ASSETS CASH On Hand—Petty fund\$ On Deposit	379.49 50,927.34	\$ 51,306.83
SECURITIES U. S. Government obligations	-11/1	

\$197,140.78

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234	TRANSACTIONS OF THE A. S. I	М.	December
ess reserve .	st	\$172,140.78 1,882.64	174,023.42
	vable	\$ 9,780.58 949.08	8,831.50
INVENTO Sound and unb less reserve	ORY ound publications, books, paper stock, etc		25,028.59
OTHER A	SSETS rees and miscellaneous accounts, etc		9,021.60
OFFICE I	FURNITURE, FIXTURES AND EQUIPMENT ision for depreciation)		7,475.80
Prepaid METAL Prepaid Wester	ED convention expense Progress publication expense rn Metal Show expense rance premiums, etc.	5,572.89 2,309.09	26,082.41
			\$301,770.15
4			7.5-41.70.13
LIABILIT	TIES AND SURPLUS		\$ 6,037.78
eserves	• • • • • • • • • • • • • • • • • • • •		45,000.00
dvance conver	ED INCOME ntion receipts		48,467.50 202,264.87
			\$301,770.15
	m Balance Sheet prepared by Ernst & Ernst, Cer	tified Public	Accountants.
	INCOME AND EXPENSE		Accountants.
	INCOME AND EXPENSE AMERICAN SOCIETY for META		Accountants.
	INCOME AND EXPENSE AMERICAN SOCIETY for META For the year ended August 31, 1937		Accountants.
INCO METAL PRO Advertising Subscription Advertisers Reprints	INCOME AND EXPENSE AMERICAN SOCIETY for META For the year ended August 31, 1937		Accountants. \$105,640.68
INCO METAL PRO Advertising Subscription Advertisers Reprints Sales MEMBERSHI Membershi	INCOME AND EXPENSE AMERICAN SOCIETY for META For the year ended August 31, 1937 ME OGRESS"—MONTHLY PUBLICATION S' Service	\$ 98,273.75 2,590.59 2,423.55 2,212.04	
INCO METAL PRO Advertising Subscription Advertisers Reprints Sales MEMBERSHI Membershi Sustaining	INCOME AND EXPENSE AMERICAN SOCIETY for META For the year ended August 31, 1937 ME OGRESS"—MONTHLY PUBLICATION S' Service PS pp dues	\$ 98,273.75 2,590.59 2,423.55 2,212.04 140.75	\$105,640.68
INCO METAL PRO Advertising Subscription Advertisers Reprints Sales MEMBERSHI Membershi Sustaining Less appor	INCOME AND EXPENSE AMERICAN SOCIETY for META For the year ended August 31, 1937 ME OGRESS"—MONTHLY PUBLICATION Some Some Some Some Some Some Some Som	\$ 98,273.75 2,590.59 2,423.55 2,212.04 140.75	\$105,640.68 , 65,856.71
INCO METAL PRO Advertising Subscription Advertisers Reprints Sales MEMBERSHI Membershi Sustaining Less apport 936 CONVEN Space rent Other inco NATIONAL I Advertisin	INCOME AND EXPENSE AMERICAN SOCIETY for META For the year ended August 31, 1937 ME OGRESS"—MONTHLY PUBLICATION g ms s' Service PS ip dues	\$ 98,273.75 2,590.59 2,423.55 2,212.04 140.75 \$106,354.27 40,497.56 \$ 65,353.00	\$105,640.68 . 65,856.71 72,706.74
INCO METAL PRO Advertising Subscription Advertisers Reprints Sales MEMBERSHI Membershi Sustaining Less apport 936 CONVEN Space rent Other inco NATIONAL I Advertising Sales RANSACTIONS- Profit on disponits on disponits earns	INCOME AND EXPENSE AMERICAN SOCIETY for META For the year ended August 31, 1937 ME OGRESS"—MONTHLY PUBLICATION g ms s' Service PS p dues exhibitors' dues tionment of dues to chapters NTION—CLEVELAND tals me METALS HANDBOOK g and cuts d and purchased for resale ividends bound copies, subscriptions, etc. sal of securities—net	\$ 98,273.75 2,590.59 2,423.55 2,212.04 140.75 \$106,354.27 40,497.56 \$ 65,353.00 7,353.74 \$ 11,214.65	\$105,640.68 , 65,856.71 72,706.74 20,480.51 16,352.99 6,425.17 5,251.58 2,112.39 1,710.71 434.77
INCO METAL PRO Advertising Subscription Advertising Advertisers Reprints Sales MEMBERSHI Membershi Sustaining Less apport Other inco NATIONAL In Advertising Sales Rooks published Interest and different and different and disponition of disponiti	INCOME AND EXPENSE AMERICAN SOCIETY for META For the year ended August 31, 1937 ME OGRESS"—MONTHLY PUBLICATION ST. Service PS St. dues	\$ 98,273.75 2,590.59 2,423.55 2,212.04 140.75 \$106,354.27 40,497.56 \$ 65,353.00 7,353.74 \$ 11,214.65	\$105,640.68 . 65,856.71 72,706.74

TOTAL INCOME

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Provision Loss on Lecture Books for Pencils,

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2.99 5.17 1.58 2.39 0.71 4.77 9.46 9.97 6.35

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184.37

53.72

EXPENSE			
AS SHOWN BY EXHIBIT			
METAL PROGRESS-monthly publication\$	96,204.21		
1936 Convention—Cleveland	56,239.34		
National Metals Handbook	25,323.67		
Book published and purchased for resale	8,746.64		
TRANSACTIONS—bound copies, subscriptions, etc	18,487.42		
General reprints	57.86		
THE REVIEW	4,549.74		
General expense	10,593.58		
Secretary's office	11,266.12		
Chapters	7,481.56		
Accounting department	6,124.19		
Directors' expense	1,947.41		
President's expense	1,460.02		
National committees	1,182.18	\$249,663.94	
Provision for inventory obsolescence		1,000.00	
Provision for doubtful accounts		481.25	
Loss on disposal of restricted bank account		439.97	
Lectures, medals, etc		373.93	
m 4 6 419			

The above from Income and Expense sheet prepared by Ernst & Ernst, Certified Public Accountants.

Books for library

Pencils, buttons, etc.

ANNUAL REPORT OF THE SECRETARY

October 20, 1937

W. H. EISENMAN, Secretary

The American Society for Metals, on October 1, 1937, had a total membership of 10,028. Of this number 8428 or 84.5 per cent were the membership classification; 591 or 5.9 per cent were sustaining members; while 850 or 8.5 per cent were juniors.

On October 1, 1936, the Society had a total membership of 7691. This shows an increase for the year of 2337 members, or an increase of 30 per cent. This represents the largest increase in membership in any one year of the Society.

This represents not only the largest number of new members secured in any one year, but the largest percentage of increase, and is approximately two and one-half times the membership in 1932.

An interesting sidelight on the membership of the Society and its relationship to the report of the Treasurer is that the total expenditures of the Society for the past year were approximately \$291,000. If one divides that amount by the average number of members during the past year, 9000, you will observe that the expenditure per member has been \$32.30, of which amount only

MEMBERSHIP 1920-1937

Year	· - '.	Total Members	Increase		Per Cent Increase
1920		1724			
1921	-	2168	444		25.7
1922		2313	145		6.7
1923		2746	433		18.7
1924		3026	280		10.2
1925		3363	337		11.1
1926		4000	637		18.9
1927		4653	653	-	16.3
1928		4844	191		4.1
1929		5615	771		16.0
1930		6138	523		9.3
1931		5761	-377		-6.15)
1932		4468	-1293		-22.4 \
1933		4470	2		0.045
1934		5443	973		21.8
1935		6880	1437		26.4
1936	(7691	811	and.	11.8
1937		10,028	2337		30.3

\$10.00 represents the member's dues. The additional \$22.30 has been obtained from activities of the Society other than dues and shows how important the other activities of the Society are if the Society is to continue to make an expenditure of \$32.30 for every \$10.00 received as dues.

Publication Committee

The Publication Committee for the year 1936 was made up of the following: A. A. Bates, Chairman; H. E. Ardahl, John Chipman, Earnshaw Cook, E. S. Davenport, C. N. Dawe, M. A. Grossmann, W. E. Harvey, L. W. Kempf, V. N. Krivobok, D. F. McFarland, O. W. McMullan, N. I. Stotz, Joseph Winlock and Ray T. Bayless, Secretary.

Throughout the year and up to the present time the Committee has carefully reviewed and approved 42 papers, rejected 3, and is in the process of reviewing 6 papers at the present time. Four papers were withdrawn by their authors.

The last meeting of the Publication Committee was held in Cleveland at the National Offices during the day of December 18, 1936. Those in attendance were: A. A. Bates, Chairman, E. S. Davenport, W. E. Harvey, O. W. McMullan, N. I. Stotz, Joseph Winlock, John Chipman, Earnshaw Cook, L. W. Kempf, V. N. Krivobok, Ray T. Bayless, Secretary, E. C. Bain, President, and W. H. Eisenman, Secretary A.S.M. The following was made a part of the record of this Publication Committee meeting:

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k, k, nIn accordance with the resolution of the Publication Committee at its meeting of June 19, 1936, the Secretary reported the appointment by the then Chairman W. P. Sykes of Messrs. A. A. Bates, H. W. McQuaid, M. A. Grossmann, O. W. McMullan, H. B. Knowlton and Ray T. Bayless, members of a committee to arrange for the Symposium on Carburizing for the 1937 Convention to be held in Atlantic City. The first meeting of this symposium committee was arranged for and held in Chicago December 28, 1936. The work of this committee is evidenced by the splendid program of papers presented at this Convention on Thursday and Friday with four sessions devoted to the subject of the carburizing of steel.

The Publication Committee selected the topic of "Alloying Elements in Medium and Low Alloy Steels" as the subject matter for the symposium program for 1938 to be held at the A.S.M. Convention next year in Detroit. The work on this program is now under way.

Transactions

There have been published, since the last annual meeting of the Society, four quarterly issues of Transactions, i.e., December 1936, March, June and September 1937. The number of pages published in these four issues totals 1276, constituting 44 papers.

The Transactions published since December 1936 contain papers presented at the 1936 Convention together with others received during the year. The last issue of Volume 24 (December 1936) contained 9 papers and the first three issues of Volume 25 for 1937 contained 35 papers with discussions.

Metal Progress

METAL PROGRESS continues to be an outstandingly successful purveyor of information to the members, and has attained that status as a valuable asset to the American Society for Metals which was envisioned at its foundation seven years ago. An attractive net profit is shown on the balance sheet even though production expenses have increased during the last 12 months, not only because of increased unit costs for paper and printing, and the larger size of the magazine, but more especially because of the increased circulation to the expanding membership. Comparative figures illustrate the situation:

Nov.	1036	to Oct	1937	Nov	1935	to Oct	1936
LYOV.	1230.	IU VICE.	1237	LVUV.	1200.	IU CICL	1220

Net editorial pages	641	607
Net advertising pages	920	708
Total copies printed126	5,400	101,200

To counteract these largely increased costs of mechanical produc-

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tion, reductions have been made in expenses. This would have been insufficient had there not been the gratifying increase in advertising volume mentioned above, namely 920 pages for the last 12 months as compared with 708 pages for the preceding period—an increase in space of almost exactly 30 per cent.

One has no hesitancy in stating that the Society approves of METAL PROGRESS. During the last year, President Bain and your Secretary visited nearly all the Chapters of the Society and in each instance, when meeting with the local executive committee, asked for the frankest criticism of the magazine. A number of valuable suggestions were made, which in practically all cases had to do with improvements in detail. The consensus was distinctly one of approval.

The Editorial Advisory Board of METAL PROGRESS has given careful consideration to ways and means of improving the magazine to make it more and more useful to a constantly increasing percentage of our members.

Preprints

Fifty papers were presented before the 18th annual Convention held in Cleveland last year (1936). For this year's Convention 43 papers have been scheduled for presentation, 28 of which have been preprinted and distributed to those members requesting them. The total number of pages of preprints for 1936 was 933; the total number of preprint pages for 1937 is 563. The number of requests for preprints this year totals over 1200.

Books

Due to the large demand for the Grossmann book entitled "Principles of Heat Treatment," it was found necessary to reprint the book, but, before reprinting, it was revised by the author and issued in revised form as a second printing.

The papers that were presented at the 1936 Convention on the Symposium on the Plastic Working of Metals have been made available in cloth covered book form.

There is now in preparation for publication the series of lectures by Mr. Earnshaw Cook at the present Convention on "Open-Hearth Steel Making."

The Review

Because of the rapid growth in Society membership, the large increase in number of Chapters, and the ever-widening field of Chap-

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ter activities, it was decided in January 1937 to publish The Review on a monthly basis rather than bi-monthly, with the exception of July and September.

A "Junior Page," devoted to matters of interest to the large number of junior members and junior groups in the Society, was inaugurated with the March 1937 issue, and has proven quite popular. Another new feature this year which has received favorable comment has been the publication of a series of lectures of a fundamental nature on "Behavior of Metals in Service," which was given as an educational course by the Philadelphia Chapter last winter.

From November 1936 to date (nine issues) 3699 column-inches or 81 per cent was devoted to editorial matter, 863 column-inches or 19 per cent to advertising, and 1481 or 32 per cent of the total space to reports of Chapter meetings.

Metals Handbook Committee

Metals Handbook Committee has had a very active year making plans for the preparation of the next edition of the Handbook.

The first meeting was held in the National Offices of the Society and consisted of a two-day meeting on February 12 and 13, 1937. The second meeting was also held in the National Offices on October 1 and the third meeting for the year is being held this week on Thursday at the Ambassador Hotel. The personnel of the committee is as follows:

J. P. Gill, Chairman	R. B. Schenck
R. L. Dowdell	K. R. Van Horn
S. L. Hoyt	Jerome Strauss
R. H. Aborn	H. L. Maxwell
G. C. Riegel	J. H. Hall
A. M. Steever	C. W. Obert

Mr. W. C. Chancellor was a member of the committee until his death last March.

The committee has prepared an extensive program for new articles and reports for the next edition of the Handbook.

The following 34 subcommittees have been organized and assigned Handbook problems:

Subcommittee on Aging		
Subcommittee on Alloying Elements	1.1	
Subcommittee on Carburizing		
Subcommittee on Case Depth Measurement		
Subcommittee on Cast Heat Resisting Alloys		
Subcommittee on Cold Shearing of Steel Bars and Billets		
Subcommittee on Determination of Oxygen, Hydrogen, and	Nitrogen	iı
Carbon Steels		

Subcommittee on Electrical Resistance Alloys

Subcommittee on Forgings

Subcommittee on Hardness Testing

Subcommittee on Heat Treatment of Steel Castings

Subcommittee on Impact Testing Subcommittee on Definitions

Subcommittee on Identification of Nonmetallic Inclusions by Use of Polarized Light

Subcommittee on Significance and Interpretation of Nonmetallic Inclusions

Subcommittee on Linear Expansion of Steels

Subcommittee on Machinability

Subcommittee on Mechanical Factors Affecting the Machinability of Steel Subcommittee on Cutting Fluids as they Affect the Machinability of Steel Subcommittee on Factors Affecting Machinability of Various Steels

Subcommittee on Macro Etching

Subcommittee on Mechanical Property Charts Subcommittee on Magnetic Testing

Subcommittee on Metallic Coatings for Ferrous Metals

Subcommittee on Notch Bar Testing Subcommittee on Physical Constants

Subcommittee on Pyrometry

Subcommittee on Quenching Media

Subcommittee on Rolls Subcommittee on Salt Baths Subcommittee on Strip Subcommittee on Tool Steels

Subcommittee on Welding and Cutting

Subcommittee on Wrought Heat Resisting Alloys

In addition to the above subcommittees the following 19 subjects have been assigned to authors to prepare articles for the Handbook:

Corrosion Fatigue Fatigue Testing Fluidity of Metals

Fuel Oils

Furnace Atmospheres High Strength Low Alloy Steels

Induction Hardening

Magnetically Soft Materials

Oil-less Bearing from Standpoint of Powder Metallurgy

Photomicrography

Physical Properties of Large Sections

Powder Metallurgy Plastic Refractories

Preparation of Metallographic Specimens

Recrystallization of Metals Shaping and Forming of Metals (A series of articles)

Statistical Analysis of Test Results and Probability Distribution Curves

Stainless Steel Numbering System

X-Ray Diffraction and Crystal Structure of Metals

After working out the program for new articles, the next important project for the committee was to make arrangements for making revisions in the present articles in the Handbook for the next edition. The committee has prepared plans for this work and by the last of

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last of this year all former authors and subcommittees will be contacted and requested to revise their articles and reports for the 1938 Handbook.

Nonferrous Data Sheet Committee

The Nonferrous Data Sheet Committee of the Institute of Metals Division of the American Institute of Mining and Metallurgical Engineers, through a co-operative arrangement with the American Society for Metals, has again prepared a very comprehensive program for the nonferrous section of the Handbook. The personnel of the Nonferrous Data Sheet Committee in charge of this program is as follows:

> Jerome Strauss, Chairman E. E. Schumacher W. L. Fink

W. E. Remmers Lyall Zickrick E. M. Wise

The program for new articles to be prepared by the Nonferrous Data Sheet Committee is as follows:

Hot Plastic Working of Nonferrous Metals

Properties of Cadmium-Copper-Silver Alloys Constitution of Cadmium-Silver Alloys Constitution of Magnesium-Aluminum-Zinc Alloys

Constitution of Zinc-Silver Alloys

Constitution of Zinc-Manganese Alloys

Polishing of Zinc and its Alloys for Metallographic Examination Etching Solutions for Zinc and its Alloys for Metallographic Examination Physical Constants of Nickel

Properties of Commercially Pure Nickel Welding of Aluminum and its Alloys

Protection of Aluminum and its Alloys against Corrosion

Soldering of Aluminum and its Alloys Properties of Lead-Calcium Alloys

Properties and Uses of Gold Alloys

Properties of Lead-Antimony-Tin Alloys Selection of Alloys for Bearing Purposes Physical Constants of Lithium

Constitution of Copper-Zinc-Tin Alloys

The Phase Rule (general article)
Properties of Cast Manganese Bronzes
Properties of Wrought Manganese Bronzes
Articles on Die Casting Tin, Lead, Copper and their Alloys

Properties of Aluminum Alloy 27S

Heat Treatment of Cast Aluminum Alloys

Properties of Aluminum Alloys 25S Properties of Wrought 60-40 Brass Properties of Wrought 80-20 Brass

Properties of Cast Bronzes

Properties of Magnesium Forging Alloys

Properties of Wrought Magnesium Alloys (1.5% min.) Properties of Wrought Magnesium Alloys (4% aluminum)

Distribution of 1936 Metals Handbook

The 1936 edition of Metals Handbook was published just twelve months ago and consisted of 13,000 copies. Because of the growth of the Society and requests for the Handbook from the metal industry, the Society has in stock 1671 copies.

The Handbook has developed so extensively in value and usefulness that this issue has received an enthusiastic reception from the Society members and the metal industries. The book is the result of the efforts and co-operation of about 2500 members of the Society who have contributed their time and information which have made Metals Handbook available for all the members. To these men the Society is indebted and wishes to express its thanks and appreciation.

A number of Chapters of the Society located within convenient distances of one another have established a very estimable and desirable activity of holding sectional meetings.

All of these meetings held during the past year have achieved splendid success, not only in the development of fellowship but in establishing a greater community interest and sphere of influence. Sectional meetings in 1937 were held in the following places:

New England-at New Haven, May 21; Philadelphia, Lehigh Valley, New York, New Jersey-at Newark, May 14; Southern Tier, Syracuse, Rochester—at University of Cornell, April 19; Columbus, Cincinnati, Dayton—at Columbus, April 28.

Last year the Board of Trustees invited the six Secretaries of Chapters who had served ten years or more in that capacity to be the guests of the Society for the week of the convention. They were Howard Handy of Boston, Irving Matthews of Rochester, C. G. Peterson of Rhode Island, H. L. Walker of Pittsburgh, Alexis Caswell of North West, and T. N. Holden of New York.

It is a distinct pleasure and a marked privilege for the Society to recognize this year the long-standing services of Fred Reiter, industrial engineer of the Dayton Power & Light Co., Dayton, Ohio, who is completing ten years of continuous service as secretary of the Dayton Chapter. He is attending this convention as the honored guest of the Society.

Metal Exposition

This year's exposition is not only the most attractive but again the largest metal show in American Society for Metals' history. In only one year has the total space reservation exceeded that of 1937 and tha try acc

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and that was in Detroit in 1927 when another unit of the metal industry accounted for approximately one-third of the space reserved.

There are 237 exhibitors as compared to 217 last year, and they are now ensconced in as perfect an exhibition hall as there is to be found in America. It is ideal in every way because it permits all exhibitors having splendid locations on the main floor.

An exposition is a recognized method of trade promotion and business obtaining. There is no other form of advertising which brings the consumer so intimately in touch with the advertiser's product at a time when he is so willing to observe and to learn.

The National Metal Congress and the National Metal Exposition are recognized as the outstanding educational and merchandising events during any current year.

The 1938 National Metal Congress and Exposition will be held in Detroit the week of October 17, 1938.

The achievements of the American Society for Metals are one of the outstanding phenomena of the last two decades. Beginning in a very small way with a handful of interested men having a vision of useful service to the metal industry and willing to work incessantly to accomplish that worthy objective, the Society's progress along the pathway of success has been steady and continuous.

This working for the A.S.M has been a glorious interest—for the progress has been so perceptible. The workers' combined efforts have produced remarkable results, as combined efforts and teamwork always do.

The Board of Trustees honors the national committees, the Chapter officers, the authors, the industrial firms, and the many others of this successful army of co-operation in the service of their fellow metal men.

These individuals typify the success of the A.S.M. because they represent the strong links in the chain of earnest laborers who have given so copiously for the good of the cause.

These members would no more think of permitting these staunch links to lay aside their share of the work than they would permit general disintegration of the aims and purposes for which the Society was established.

And so we will pull down the curtain on this, the nineteenth year, of the American Society for Metals. It is placed in the archives of advancement—and remains as a record of the metal progress of the period, as well as a pleasant memory.

This report is a valedictory message to the year just closed—but at the same instant is a salutatory greeting given to the hardworking, highly esteemed and successful members of the A.S.M., envisioning another year of faithful and conscientious service—heralding the beginning of even greater achievements.

ELECTION OF OFFICERS

EDGAR C. BAIN, President

Complying with the Constitution I appointed as of March 15th the following Nominating Committee, selected from the list of suggested candidates received prior to March 1, 1937:

G. V. Luerssen (Chairman),
Carl G. Johnson,
Irving C. Matthews,
Edward G. Mahin,
J. M. Watson,
A. B. Beaver,
Kenneth H. Hobbie,

Lehigh Chapter
Worcester Chapter
Rochester Chapter
Notre Dame Chapter
Detroit Chapter
Dayton Chapter
Chicago Chapter

This Committee met in Pittsburgh on May 20, 1937, and made the following nominations:

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	President	George B. Waterhouse, Professor of Metallurgy, Massachusetts Institute of Technology.		
	Vice President	William P. Woodside,	Vice President, Climax Molybdenum Company, Detroit.	
	Treasurer (2 years)	Bradley Stoughton,	Dean of Engineering Dept., Lehigh University, Bethlehem, Pa.	
	Trustees (2 years)	James P. Gill,	Chief Metallurgist, Vanadium-Alloys Steel Co., Latrobe, Pa.	
		Harvey A. Anderson,	Metallurgical Engineer, Western Electric Co., Chicago.	

A report of these nominations duly appeared in The Review for May, 1937.

I have been informed by the Secretary that no additional nominations were received prior to July 15, 1937, for any of the vacancies occurring on the Board of Trustees. Consequently the nominations were closed. I shall now call upon the Secretary to carry out the provisions of the Constitution in respect to the election of National Officers. (Secretary casts ballot)

The provisions of the Constitution having been complied with I hereby declare the candidates heretofore named to be duly and unanimously elected to the several specified offices, the terms of each officer just elected beginning on the day following the close of this annual meeting.

Statement of the ownership, management, circulation, etc., required by the acts of congress of August 24, 1912, and March 3, 1933, of Transacrious of American Society for Metals, published quarterly at Cleveland, Ohio, for October 1, 1937, State of Ohio, county of Cuyahoga, ss. Before me, a notary public, in and for the State and county aforesaid, personally appeared Ray T. Bayless, who, having been duly sworn according to law, deposes and says that he is the editor of the Transactions of the American Society for Metals, and that the following is, to the best of his knowledge and belief, a true statement of the ownership, management, etc., of the aforesaid publication for the date shown in the above caption, required by the Act of August 24, 1912, as amended by the Act of March 3, 1933, embodied in section 537, Postal Laws and Regulations to wit

1.—That the names and addresses of the publisher, editor, managing editor, and business managers are: Publisher, American Society for Metals, 7016 Euclid Ave., Cleveland, O.; Editor and Managing Editor, Ray T. Bayless, 7016 Euclid Ave., Cleveland, O.; Business Manager, W. H. Eisenman, 7016 Euclid Ave., Cleveland, O.

2.—That the owner is: The American Society for Metals,

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7016 Euclid Ave., Cleveland, Ohio, which is an educational institution, the officers being, President, E. C. Bain; Vice President, G. B. Waterhouse; Treasurer, W. P. Woodside; Secretary, W. H. Eisenman; Trustees: R. S. Archer, O. W. Ellis, R. L. Wilson, S. C. Spalding, R. L. Kenyon. All officers

as above, 7016 Euclid Ave., Cleveland, Ohio.

3.—That the known bondholders, mortgagees, and other security holders owning or holding 1 per cent or more of total

amount of bonds, mortgages, or other securities are: none.
4.—That the two paragraphs next above, giving the names of the owners, stockholders, and security holders, if any, contain not only the list of stockholders and security holders as they appear upon the books of the company but also, in cases where the stockholder or security holder appears upon the books of the company as trustee or in any other fiduciary relation, the name of the person or corporation for whom such trustee is acting, is given; also that the said two paragraphs contain statements embracing affiant's full knowledge and belief as to the circumstances and conditions under which stockholders and security holders who do not appear upon the books of the company as trustees, hold stock and securities in a capacity other than that of a bona fide owner; and this affiant has no reason to believe that any other person, association, or corporation has any interest direct or indirect in the said stock. bonds, or other securities than as so stated by him. Ray T. Bayless, managing editor, sworn to and subscribed before me this 1st day of Octobe

(Seal) Arthur T. Wehrle, notary public. (My commission expires January 21, 1938.) Metallography Progresses - FISHER

Scientific Company designs and builds modern laboratory appliances for the preparation of metal specimens for examination under magnification.

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12-266 Metallographic Pelisher. Fisher. (left)
This small polisher has two speeds, 550 and 1100
r.p.m. with full power and strong torques. The polisher has a cast fron housing, aluminum bowl in two parts, 6-linch head and counterpoised locking ring. Each, \$120.00





2-545 Metallographic Emery Papers (left). Distinguished for their even, uniform surface. Grade Nos. 0000, 000 and 90 are recommended for inel grinding. Grade Nos. 0, 1, 1G and 2 (increasing coarseness) are recommended for preliminary strinding.

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Grinder, Motor Driven. (left). For rapidly surfacing metal specimens. Employs 4-inch belts. Adjustable rest holds specimen on helt. Each, \$113.00

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Grade No. 2, for medium hard matels med as brade and popper. Per ounce, \$1.40

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